

STUDIES ON CERTAIN ASPECTS OF SHRINKAGE AND SWELLING
IN CLAY-SAND SYSTEMS

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CONTENTS

	ACKNOWLEDGEMENTS	iii
	ABSTRACT	iv
CHAPTER 1	INTRODUCTION	1
CHAPTER 2	SHRINKAGE AND SWELLING IN CLAY - A CRITICAL REVIEW	3
	2.1 Clay mineral structure	
	2.2 Measurement of swelling and shrinkage properties	
	2.3 Prediction of magnitude of swelling	
	2.4 Prediction of shrinkage	
CHAPTER 3	ROLE OF PARTICLE SIZE AND PACKING IN MATERIAL SYSTEMS	18
CHAPTER 4	SWELLING CHARACTERISTICS OF CLAY-SAND SYSTEM	21
	4.1 Experimental investigation	
	4.2 Observations	
	4.3 Discussion	
CHAPTER 5	SHRINKAGE CHARACTERISTICS OF CLAY-SAND SYSTEMS	26
	5.1 Experimental investigation	
	5.2 Results	
	5.3 Discussion	
	5.4 Influence of grain size of participating sand in the clay-sand mixes	

CHAPTER 6	X-RAY INVESTIGATION OF SHRINKAGE PROCESS IN BENTONITE	38
	6.1 Instrumental setup	
	6.2 Experimental investigation	
	6.3 Results and discussion	
	6.4 Summary	
CHAPTER 7	SYNTHESIS	48
	REFERENCES	52
	APPENDIX	63

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ABSTRACT

The shrinkage properties of the clay-sand systems have been examined. It has been observed that the shrinkage limit variation with increasing percentage of clay in the mix for any particular grain size of sand takes place in two rates - one rate up to a critical packed situation till which state all the clay particles occupy the available void space within the granular particles and at another rate beyond this critical state wherein the clay particles establish continuity. As such it is established by the present study that for montmorillonite and kaolinite with sand, the rate of variation of shrinkage limit depends upon the state of existence of clay particles in the mix (as to whether they are in a continuous or discontinuous state). The influence of clay percentage and grain size of sand on the shrinkage limit, shrinkage index and the swelling characteristics of these clay-sand mixes have been studied. The increase in shrinkage index has a tendency to increase the swelling activity of the clay-sand mix. With finer grain size of sand in the mix, the critical packing limit is attained earlier and the shrinkage index tends to increase slightly.

X-ray diffraction studies have also been undertaken for the dehydration of pure bentonite and bentonite-sand systems and the corresponding lattice variation with time has been reported.

CHAPTER 1

INTRODUCTION

Expansive soils have been causing difficult problems in the field of civil engineering. Light building structures, highways, canal linings and many other structures which are founded on or built with expansive soils undergo severe shrinking and swelling. In many parts of India for example, Madhya Pradesh, Andhra Pradesh, parts of South India and certain areas of Orissa, Bihar and West Bengal, such expansive soils are frequently met with and cause serious engineering problems.

It is of vital importance to study the intrinsic properties in connection with the engineering behaviour of such soil to generalise and predict some of its behaviours so that necessary steps can be incorporated into the design and construction of structures to be built over such expansive soil. In soil mechanics the fraction of a soil which is less than two microns is designated as clay and is considered to be mainly responsible for the physical and chemical behaviour which the soil exhibits owing to the colloidal state of existence of the particles.

In chapter 2 a critical review of available information on the swelling and shrinkage of clay has been given. In chapter 3 the role of particle size and packing in a material system in general has been discussed. Their impact

on the shrinkage and density characteristics of a sand clay system has been brought about through experimental studies made on the swelling and shrinkage characteristics of clay sand system and discussed in chapters 4 and 5. An attempt has been made to investigate the shrinkage characteristics of clay sand mixture with time by x-ray diffraction studies in chapter 6. Lastly chapter 7 gives a summary of all the work done and brings out few critical conclusions.

The clay selected for the present study is a Bentonite clay from Wyoming, U.S.A., and the sand selected is a local sand obtained from sand pits near Kalpi, Uttar Pradesh.

SHRINKAGE AND SWELLING IN CLAY - A CRITICAL REVIEW

2.1 Clay mineral structure

The shrinkage and swelling properties of clays are a consequence of the structure of clays with the specific charge distribution in the lattice. In general all the clay minerals are composed of alternate layers^{of} silica tetrahedra and alumina octahedra. In the silica tetrahedra the silicon atom is equidistant from four oxygens, or hydroxyls if needed to balance the structure, arranged in the form of a tetrahedron with a silicon atom at the centre. The tetrahedra are so arranged that their tips point towards the same direction with the bases forming a hexagonal network (fig. 2.1). The thickness of the unit is 4.93 \AA in clay minerals. In the octahedral layer aluminum, iron or magnesium atoms are in octahedral coordination with six oxygens or hydroxyls (fig. 2.1). When aluminum is present, only two thirds of the possible positions are filled to balance the structure (Gibbsite layer). When magnesium is present all the positions are filled to balance the structure (Brucite layer). The thickness of the octahedral unit is 5.05 \AA in clays.

In kaolinite, the unit layer is composed of a single silica tetrahedra sheet and a single aluminum octahedral sheet combined in a unit so that the tips of the silica tetrahedra and one of the layers of the octahedral sheet form a common layer. The tips of the tetrahedra point towards the

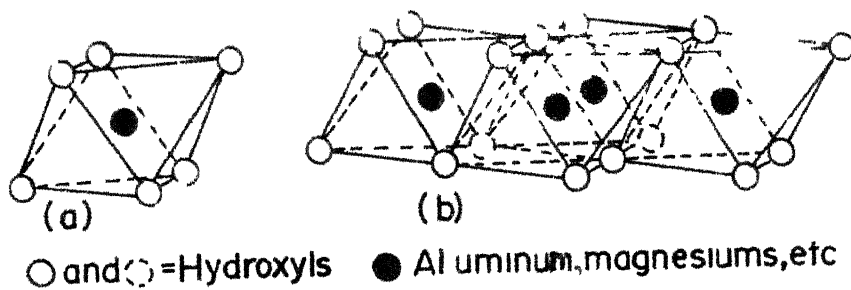


FIG.2(a) DIAGRAMMATIC SKETCH SHOWING (a) SINGLE OCTAHEDRAL UNIT AND (b) THE SHEET STRUCTURE OF THE OCTAHEDRAL UNIT

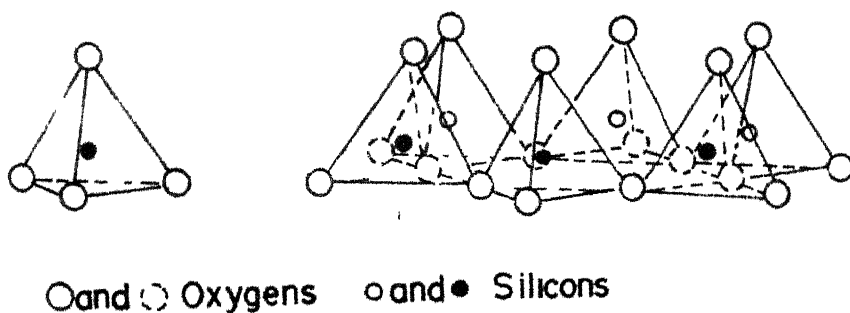


FIG.2(b) DIAGRAMMATIC SKETCH SHOWING (a) SINGLE SILICA TETRAHEDRON AND (b) SHEET STRUCTURE OF SILICA TETRAHEDRA ARRANGED IN A HEXAGONAL NETWORK.

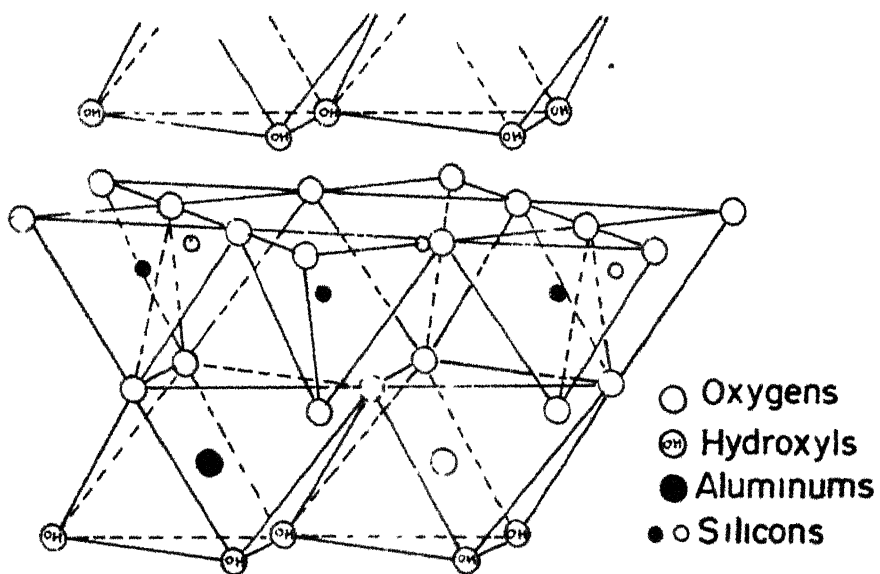
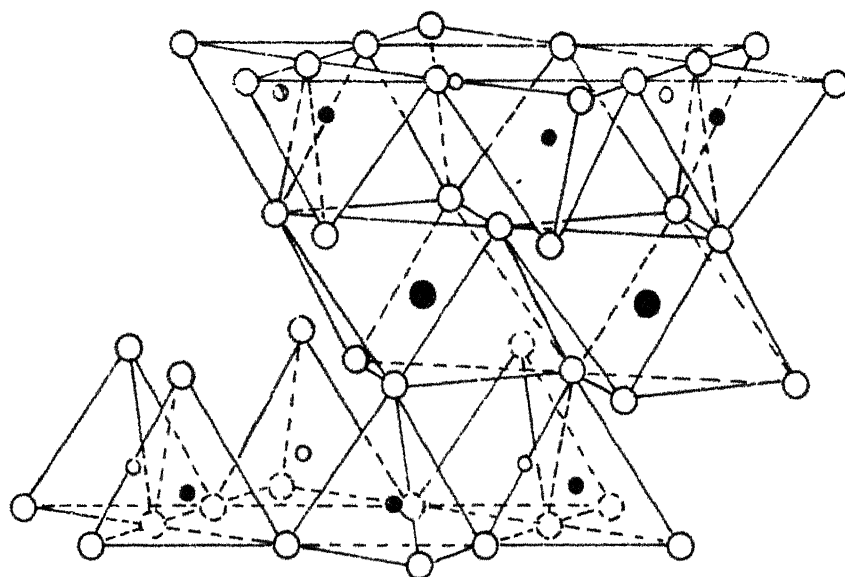
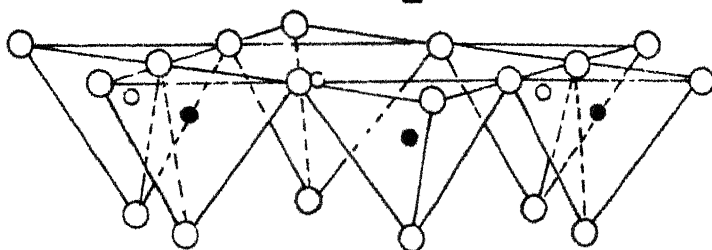


FIG.2.2 DIAGRAMMATIC SKETCH OF THE STRUCTURE OF THE KAOLINITE LAYER, AFTER GRUNER. (1932)



Exchangeable Cations
n H₂O



○ Oxygens ○ Hydroxyls ●
○ and ● Silicon, occasionally aluminum

FIG.2.3 DIAGRAMMATIC SKETCH OF THE STRUCTURE OF MONTMORILLONITE ACCORDING TO HOFMANN, ENDELL, AND WILM, MARSHALL AND HENDRICKS. (1933, 1935, 1942)

centre of the unit. In the common layer for tetrahedral and octahedral sheets two thirds of the atoms are shared by the silicon and the aluminum and then they become O instead of OH. The aluminum atoms are so placed that two aluminums are separated by an OH above and below. The atomic planes in kaolinite are

	Charges	
Si-tetrahedron	$\begin{bmatrix} 6 \text{ O}^{-2} & -12 \\ 4 \text{ Si}^{+4} & +16 \end{bmatrix}$	
Al-octahedron	$\begin{bmatrix} 4 \text{ O}^{-2} + 2(\text{OH})^{-} & -10 \\ 4 \text{ Al}^{+3} & +12 \\ 6 (\text{OH})^{-} & -6 \end{bmatrix}$	<p>- 28</p> <p>+ 28</p>

From the above distribution of charges in the structural unit of kaolinite it is evident that the charges are balanced and the kaolinite sheets are neutral. This neutral state of the kaolinite unit accounts for its least cation exchange capacity and absence of activity. A regular stacking and closed spacing of layer causes a relatively strong bond between successive layers which accounts for the absence of interlayered water.

As contrast to kaolinite montmorillonite is a three layered clay mineral. The structural unit for montmorillonite consists of a central alumina octahedral sheet sandwiched (fig.2.3) between two silica tetrahedral sheets, the tips of the tetrahedra in both the sheets pointing towards the centre of the unit. The atoms common to both the tetrahedral and the

octahedral layer become O instead of OH. The theoretical charge distribution (Carroll¹⁶, 1959) without considering substitution within the layer, is as follows:

Charges			
Si-tetrahedron	$\begin{bmatrix} 6 \text{ O}^{-2} \\ 4 \text{ Si}^{+4} \end{bmatrix}$	$\begin{bmatrix} -12 \\ +16 \end{bmatrix}$	+ 44
			layer common to tetra-
			hedral or octahedral
			sheets
Al-octahedron	$\begin{bmatrix} 4 \text{ O}^{-2} \text{ } 2(\text{OH})^{-} \\ 4 \text{ Al}^{+3} \end{bmatrix}$	$\begin{bmatrix} -10 \\ +12 \end{bmatrix}$	layer common to tetra-
			hedral or octahedral
			sheets
Si-tetrahedron	$\begin{bmatrix} 4 \text{ Si}^{+4} \\ 6 \text{ O}^{-2} \end{bmatrix}$	$\begin{bmatrix} +16 \\ -12 \end{bmatrix}$	- 44

However such a theoretical charge distribution is very rare in montmorillonite. Normally the lattice is always unbalanced by substitutions like Mg^{2+} for Al^{3+} , Al^{3+} for Si^{4+} . The unbalancing may result from substitution of ions of different valencies in both the octahedral or tetrahedral sheets. Many analysis have shown the net charge deficiency to be 0.66⁻ per unit cell. This charge deficiency corresponds to about two thirds unit per unit cell. This net charge deficiency is balanced by exchangeable cations adsorbed between the unit layers or adsorption of water or other polar molecules between the layers. Consequently the cell dimensions for montmorillonite in C direction alter during the process of balancing of structure. The taking of inter-layered water between the montmorillonite lattice is facilitated

by the poor bonding between the different layers. Roth⁶⁷ (1951) has shown that the important physical characteristics of clays composed of montmorillonite are related to the regularity or randomness of the inter-layer water layers.

Thus it is evident that the swelling in expansive clays (with montmorillonite as principal clay mineral) is due to cation exchange and/or hydration which are a consequence of the crystal structure. The crystal structure of kaolinite together with the regular stacking and a stronger bond between individual layers explains the least exchange capacity and paucity of inter-layered water for kaolinite. During dehydration in expansive clays due to the expulsion of the inter-layered water shrinking of the lattice is considerable. This accounts for greater shrinkage for clays containing montmorillonite as the dominant clay mineral as compared to the clays with kaolinite as the important constituent. A detailed account of the mechanism of swelling in clays has been reported by Finn²³, Ladd⁴¹, Low⁴⁶, Denning⁴⁶, Macewan⁴⁷, Norrish⁵⁸, Barshad⁶, Seed⁷¹, Ruiz⁶⁹, and Mackenzie⁵⁰.

2.2 Measurement of swelling and shrinkage properties

Many attempts have been made to measure the amount of swelling in an expansive clay in the laboratory. Earnest Pitcher¹⁹, Rengmark and Erriction⁶⁴, Holtz and Gibbs³⁶, Wiseman and Joseph⁸⁴, Nagarajan and Sundararaman⁵⁶, Blomquist and Portigo⁹ and Lamb and Ladd⁴⁴ have measured the vertical

swelling in soils by different methods with or without surcharge pressure. Bruzsin¹³ has measured expansion per unit mass by volumenometer. Parcher and Liu⁶⁰ have developed apparatus to measure independently the vertical and horizontal swelling. Ring⁶⁶ has measured volume change of a soil when it achieved equilibrium condition after cyclic wetting and drying.

All the soils which show high expansive nature are liable to shrink more. Measurement of the swelling of soil by any of the methods mentioned from the minimum volume of the soil to its maximum volume also indirectly gives the order of maximum range of shrinking of a soil. In attempting to predict the shrinking characteristics of a soil the knowledge of the fundamental shrinking properties like shrinkage limit, shrinkage ratio etc. is necessary. The different methods suggested by various investigators to calculate the shrinkage properties in the laboratory and in practice in different parts of the world are listed below.

- 1) Linear observation method after Tempany ⁷⁷
- 2) Bar linear shrinkage test after Heidema²⁹
- 3) Direct mercury displacement or shrinkage limit method by Akroyd³
- 4) Mercury displacement or magnification method by Puri⁶²
- 5) Gas displacement method by Mathur and Singh⁷⁵

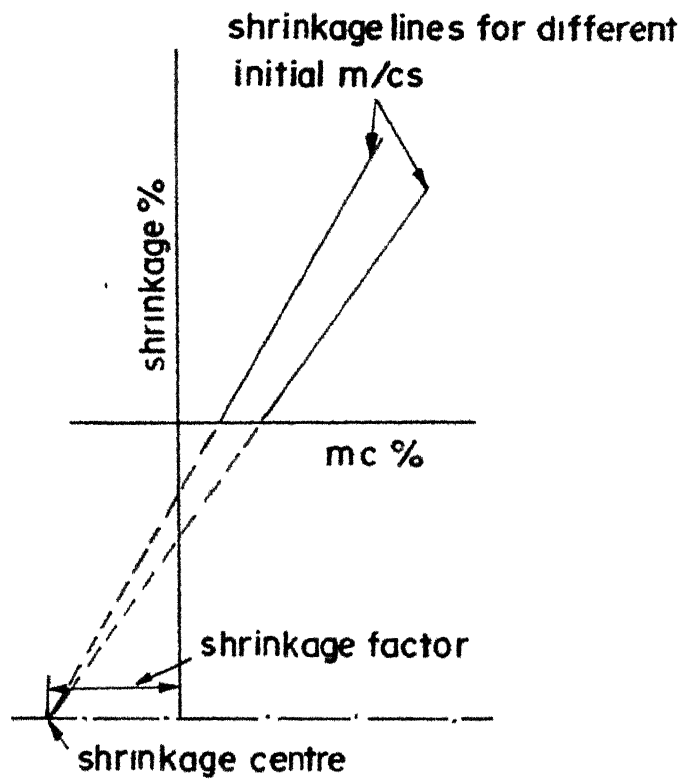


FIG.2-4 RELATIONSHIP BETWEEN SHRINKING AND m/c (AFTER HAFELI & AMBERG, 1958)

- 6) Direct displacement method a modification of A.S.T.M. procedure
- 7) Optical projection method
- 8) Consolidation method using Odeometer

Of all these methods those suitable for normal laboratory practice are:

- 1) Modified A.S.T.M.⁵⁴ method
- 2) Direct Mercury displacement method
- 3) Puri's method

Some salient points in connection with shrinkage are put here as discussed by Morgan.⁵⁵ The S.L. depends to some extent on the initial moisture content. Shrinkage limit of a soil having initial moisture content near liquid limit may differ from shrinkage limit of a soil having initial moisture content less. Care should be taken that there should be enough water initially to avoid any air bubble inside the soil. Hafeli and Amberg³⁰ noticed that the S.L. of a soil prepared at higher initial moisture content is greater than that for same soil prepared at lower moisture content. But they observed that all shrinkage lines in the 1st phase of the shrinkage process when produced back, met at a common point (fig. 2.4) called the shrinkage centre, irrespective of the initial moisture content. These investigators pointed out that in addition to shrinkage limit and shrinkage ratio the shrinkage factor which will be same for a particular soil may also be a possible indicator for the volume change characteristics of the soil.

2.3 Prediction of magnitude of swelling

The methods used for measuring the swelling of an expansive clay are time taking. So prediction of the amount of swelling out of the available physical properties of the soil is essential.

Before trying to discuss on the prediction of magnitude to which an expansive soil may swell it is necessary to know the factors which control this behaviour of the soil.

From the available literature^{18,35,42,60,71,78,84} these factors are:

- 1) Type of clay mineral and its amount in a soil
- 2) The nature of exchangeable cation in a clay-mineral structure
- 3) Dry density, initial-water content and the method of compaction
- 4) Nature of pore fluid (mainly the electrolyte concentration in it)
- 5) Test conditions like: shape and size of sample, confining conditions, temperature etc.
- 6) Present and past loading conditions on the soil
- 7) Particle size and its distribution

The most important of all these is the clay content and its type. The type of clay is taken care of by the mechanical properties of clay like liquid limit, plastic limit, shrinkage limit etc. On this basis many investigators have tried

to predict the swelling characteristics of clay in terms of these mechanical properties.

Holtz and Gibbs³⁶ present the analysis of 38 undisturbed soil samples. Out of two samples from each type of soil, placed in a consolidometer ring, one is dried in air to at least shrinkage limit and the other is allowed to swell under 1 p.s.i. loading. The difference between air dry volume and the maximum saturated volume of the sample indicates the maximum volume change.

The limits for various degree of expansion (low, medium, high and very high) as obtained by Holtz and Gibbs³⁶ for the different clay % together with the respective plasticity index and shrinkage limit are indicated in table 2.1. The soil used by them was from Friant-Kern Canals, California.

Table 2.1

Colloid content -0.001 mm	Plasticity Index	Shrink- age Limit	Estimates of pro- bable expansion (% total vol. change dry to saturated)	Degree of expansion
> 27	> 32	< 10	> 30	Very high
18 - 37	23 - 45	6 - 12	20 - 30	High
12 - 27	12 - 34	8 - 18	10 - 20	Medium
< 17	< 20	> 13	< 10	Low

- . A similar classification for the different degree of swelling is given by Altmayer¹ based on the values of linear shrinkage (table 2.2)

Table 2.2

Linear shrinkage%	Volume change
> 8	Critical
5 - 8	Marginal
< 5	Not critical

Jennings and Knight³⁸ used consolidometer to predict the total heave and the process is termed as Double Oedometer test. Simultaneously two consolidometers are used. In one the sample is saturated under specified load (0.01 T/sq') till the swelling stabilises. Then both the samples are subjected to normal loading under regular intervals to get the voidratio-log p settlement curves. Then the amount of swell is calculated as total heave minus total settlement using normal settlement theory.

Mention has already been made about Bruizn's¹³ volumeno-meter to study swelling in terms of specific expansion. Here specific expansion against moisture content is read for given confining pressure. If the specific volume changes from v_1 to v_j then the amount of heave is given by

$$H = \int_{z_2}^{z_1} \log v_j / v_1 \, dz$$

when the expansive layer extends from depth z_1 to z_2 . To perform this test the vertical and lateral confining pressures should be maintained equal to that actually existing in the field.

Lamb and Ladd⁴⁴ classify the type of swelling in a soil from the potential volume change (P.V.C) which is based on the measurement of P.I., equilibrium water content at 100% relative humidity, change in height of the sample as percentage initial height under 200 p.s.f. and the volume change from field moisture equivalent to shrinkage limit.

Table 2.3

Category of swell	P.V.C. specified	P.V.C.	H ₂₀₀	P.I.	W ₁₀₀	V _{f.m.e.} - V _{s.l.}
Non-critical	2	2	5	15	6	15
Marginal	2 - 4	4	11	25	11	24
Critical	4 - 6	6	17	35	16	33
Very critical	6					

The swelling of two similar soils may differ very much depending on the moulding water content, dry density, method of compaction, different climatic conditions to which they are subjected to and the confinement and the loading conditions under which they will swell. So Seed, Woodward and Lundgren⁷³

have adopted standardised procedure for assessing the expansion of a swelling soil. In this connection it is worthwhile to define the terms Swelling Potential and activity of the clay. The Swelling Potential is the percentage of swell of a laterally confined soil sample on soaking under 1 p.s.i. surcharge after being compacted to the maximum dry density at optimum moisture content in standard AASHTO compaction test. Activity as defined by Skempton (1953) is the rate of change of P.I. with respect to clay content. Based on the test results of S.P. for several artificially prepared soils by mixing commercial clay minerals with sand in various proportions to get many varieties of soils, it has been shown that a well-defined relationship exists between % of clay in a soil, the activity of the soil and the swelling Potential itself. The Swelling Potential is expressed in terms of activity and clay percentage as

$$S = 3.6 \times 10^{-5} A^{2.44} C^{3.44}$$

where A is activity and C is clay content in soil. For artificially prepared soils with C varying from 19 to 70 the approximate S.P. in terms of P.I. only is given as

$$S = 3.6 \times 10^{-5} \times 100 (P.I.)^{2.44}$$

According to the authors this gives value of S which deviates by $\pm 20\%$ from the value of S as calculated from the first relation. To classify a soil with respect to S.P. it is more important to categorise a soil whether it possesses high,

medium or low swelling characteristics. For this the above approach furnishes reasonable accuracy. These authors have computed a chart from the 1st equation for a wide range of soils and the chart is believed to give reasonably accurate result to serve the purpose.

It has been pointed out by Clisby¹⁴ that out of two samples one compacted at optimum moisture content and the other compacted at optimum moisture content and desiccated, the swelling potential of the desiccated sample will be more compared to the previous one. This sounds more reasonable since c.m.c. of any clayey soil will be likely on the higher side of the shrinkage limit of the soil where the soil should have a minimum volume. Earlier to this Holtz³⁵ also proposed that S.L. should be a parameter to predict swelling potential.

Satyanarayana and Ranganathan⁶³ also suggested that Shrinkage Index which they define as the difference between the liquid limit and shrinkage limit, will be a more justifiable parameter for evaluating swelling potential since a soil will have minimum volume at shrinkage limit and the difference between the L.L. and S.L. will give the direct measure of volume change of significance in engineering practice. On similar lines as Seed and others these authors derived the relation

$$S.P. = 4.57 \times 10^{-5} \times (S.A.)^{2.67} c^{3.44}$$

where S.A. is the rate of change of Shrinkage Index with

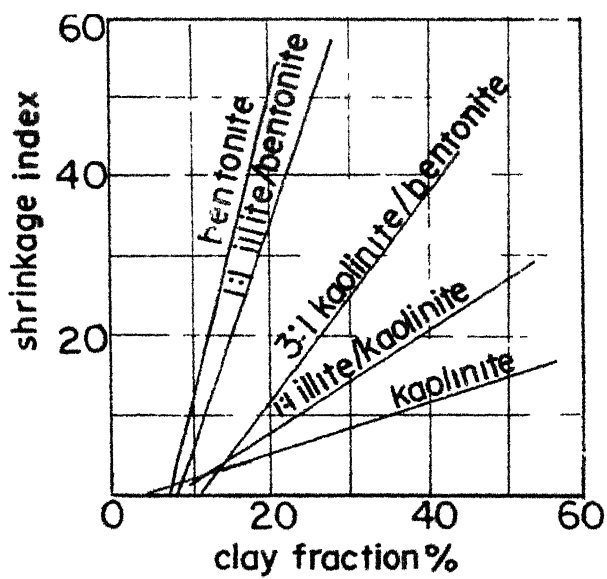


FIG. 25. SHRINKAGE INDEX CLAY-FRACTION RELATION.
(AFTER SATYANARAYANA, 1965)

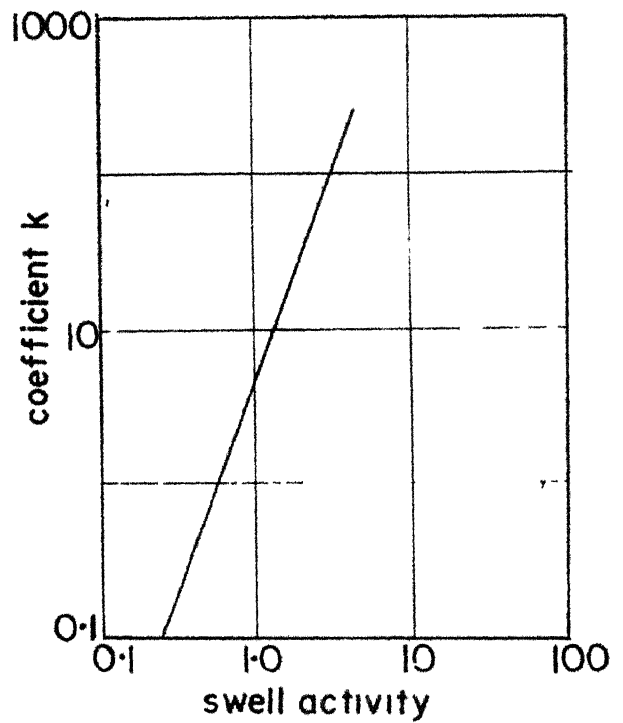


FIG. 26. RELATIONSHIP BETWEEN COEFFICIENT K & SWELL ACTIVITY FOR EXPERIMENTAL SOILS. (AFTER SATYANARYANA, 1965).

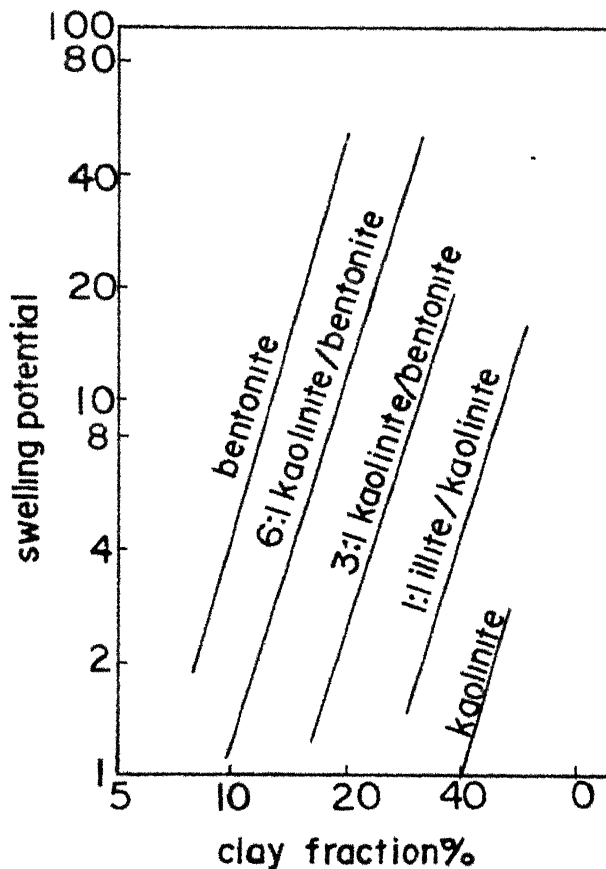


FIG. 27. RELATIONSHIP BETWEEN SWELLING POTENTIAL AND CLAY FRACTION.
(AFTER SEED ETAL, 1962)

respect to clay content. A precise derivation of the above empirical formula is presented here. From the experimental curve obtained in fig. 2.7 a relation can be presented as

$$\text{as } \text{S.P.} = K C^x$$

$$\text{or } \log \text{S.P.} = \log K + x \log C$$

where $\log K$ is intercept on the horizontal axis and x is the slope of the line observed to be a constant for any type of clay. x is found to be 3.44. Plotting K versus activity A obtained from figs. 2.7 and 2.5 it is observed that a linear relation is obtained in fig. 2.6 from where

$$K = 4.57 \times 10^{-5} (\text{S.A.})^{2.67}$$

so the expression for S.P. converts to

$$\text{S.P.} = 4.57 \times 10^{-5} \times (\text{S.A.})^{2.67} C^{3.44}$$

where $\text{S.A.} = \text{S.I.}/(C-n)$ (from fig. 2.5) n being the intercept on the horizontal axis of the S.I. - C relation. For soils included in the authors studies the average value of n was found to be 13 for soils with clay varying from 30% to 70%. For soils with clay % in this range the S.P. can be expressed in terms of S.P. only as

$$\text{S.P.}_1 = 263 \times 10^{-5} \times (\text{S.I.})^{2.67}$$

For soils with clay content lying within the same range the S.P. as reported by the authors if calculated from the above expression gives an error within $\pm 22\%$ of that calculated from the 1st expression.

W. Ring⁶⁶ has given an alternate wetting and drying procedure for evaluating shrink-swell potential and this has already been discussed before. Since this procedure also requires long time to complete Ring suggests a rapid alternate approach to predict the shrink-swell potential. He observed that a well-defined relation is maintained between shrink-swell potential and the linear shrinkage of the sample so that by performing only linear shrinkage test a reasonable approximation can be made for the required potential value of the clayey soil.

Kackar and Sengupta³⁹ have developed a useful and time saving procedure for predicting swelling potential of a clayey soil by dye adsorption method. This method can be used in the field as well as in the laboratory. The swelling studies were conducted on remoulded samples compacted by standard A.A.S.H.O. test and allowed to saturate under 1 p.s.i. surcharge. Methylene blue was used as an adsorbate in this investigation. One gram of the sample was treated with 0.2% solution of the dye with gradual intermittent stirring till it was in slight excess. The unadsorbed dye was removed and the dye adsorbed was estimated by Hilger's microptic calorimeter. A linear relation was obtained between the S.P. and the dye adsorption for each of the three types of clay - bentonite, illite and kaolinite. The relations obtained between the S.P. and the amount of dye adsorbed are:

$$\text{S.P.} = 14155 D^{2.35} \text{ for kaolinite}$$

$$\text{S.P.} = 18953 D^{2.35} \text{ for illite}$$

$$\text{S.P.} = 34577 D^{2.35} \text{ for bentonite}$$

where D is the amount of dye adsorbed by the sample. The authors have applied this procedure to field soils like black cotton soils and alluvial soils and the results are found very satisfactory. The authors report for soils below 50% clay content the dye adsorption method is very satisfactory.

2.4 Prediction for shrinkage

The lower bound of shrinking of a soil is fixed since beyond S.L. the soil does not shrink any more. So if moisture content and void ratio at any instant is known for a soil, the minimum volume it can attain can be estimated if the S.L. and shrinkage ratio values are known. So a tentative indication of a possible shrinkage movement at the surface of a soil can be obtained.

CHAPTER 3

ROLE OF PARTICLE SIZE AND PACKING IN A MATERIAL SYSTEM

The shape and size of particles, the size distribution of particles and their way of packing have got a great bearing on the physical properties of a material system. Many investigators have studied the influence of particle size and its distribution on the intensity of packing and consequently their effect on the physical properties of a mixture of materials.

In all the investigations the particles have been assumed to be ideal spheres and the effect of their size and distribution on the effective mass of the system have been studied mathematically and the theoretical conclusions have been verified for commercial mixes.

Smith⁷⁶ has calculated the minimum void volume for different types of close packings. Furnas²⁶ has considered the case of mixture of particles of different sizes where the void in the larger particles will as a rule be occupied by smaller ones and consequently be less than the voids in packings of separate components. He concluded mathematically that in a system of multiphase components the greater the size range of particles, the greater is the density. Later Hudson³⁷ and Wise⁸⁵ also reached the same conclusions. Hudson³⁷ studied the density and packing in an aggregate of mixed spheres. In a mixture which consists of equal spheres only will have an

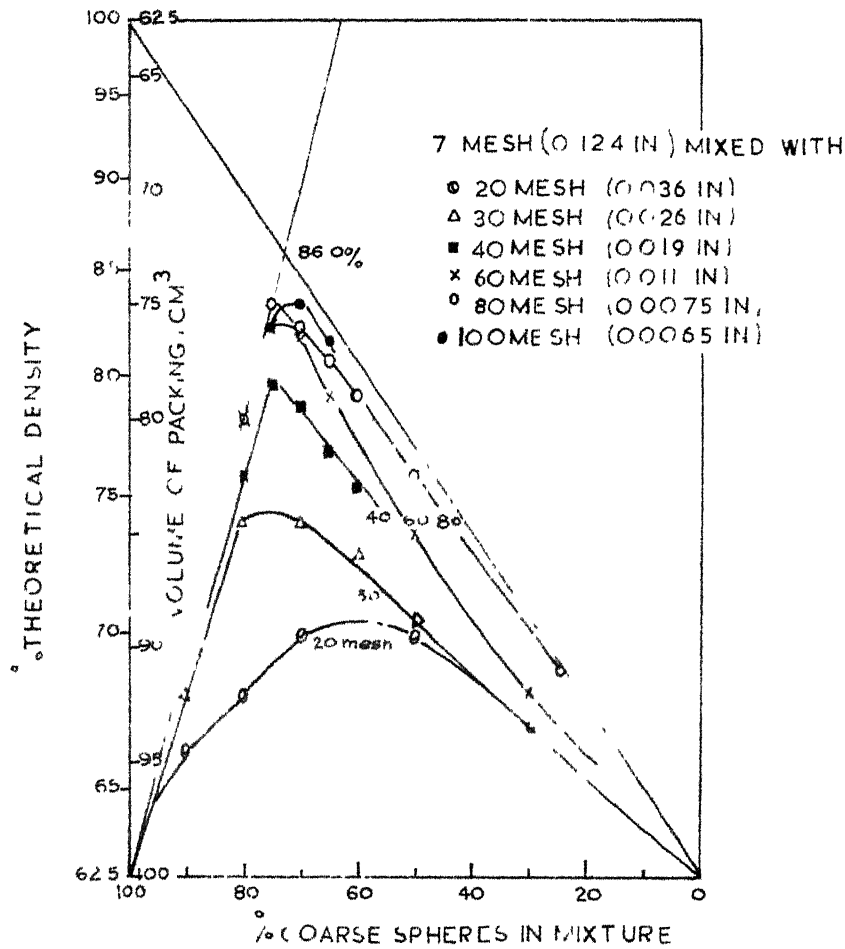


FIG 3 BINARY MECHANICAL PACKING OF COARSE STEEL SHOT WITH SOME OTHER SIZES (AFTER McGEARY, 1961)

interspace volume of about 26% at the densest packing. This has also been reported by Smith⁷⁶ Boerdijk¹⁰, that is effective density will be 0.74 which can be best attained by packing of equal spheres. In this densest state if smaller spheres can be inserted into the interspace of the bigger spheres without increasing the volume of the mix then the effective density is supposed to increase. Wise⁸⁵ has proved mathematically that the mean density of the mix can be increased over 0.74 (as mentioned earlier) if the mix will have a wide range of spheres of various sizes, at the densest packing state. McGeary⁴⁹ has conducted tests on binary and ternary homogeneous mixes and reported curves (fig. 3) for density versus percentage of coarse or finer particle in the mix. The material selected for this study was metal balls. At a densest packing attained by mechanical vibrations it was observed that peak density was attained at 20 - 40% finer material in the mix similar to that reported by the previous investigators. It was also observed that as the size of the finer material decreases the intensity of peak density increases. Hozenijk³⁴ has come to the same conclusion as Wise⁸⁵ even for heaps of spheres of limited number of radii. According to him increment in mean density is possible by random dense packing of spheres with a discrete distribution of radii.

From the above mentioned investigations it can be noticed that no study has been done to study the behaviour

of a mixture having one or more chemically active components with respect to grain size and packing. The study on variation of density and other properties should be done for such cases which are frequently met with. Some of the important conclusions drawn from the foregoing discussions are:

- 1) In a mix of particles of all equal spheres the minimum void attainable at the densest state is 26% i.e. mean density is 0.74.
- 2) When spheres of smaller size are mixed with the above system they occupy the voids inside the bigger spheres and the void reduces with increase in mean density.
- 3) In a binary mix if the finer particles are made still finer the density increases further.
- 4) In a binary mix or in a mixture of higher order if the finer sizes maintain various grading the density increases.
- 5) In a mixture of higher order the higher is the range of particle size the more is the density.
- 6) Beyond a certain percentage of finer material in a mix if it still increases then the finer materials will be in a state of continuity and the mean density of the mixture will decrease with effective increase in volume of the total mixture.

The bearing of these reports on the packing of a clay-sand mixture as observed from its shrinkage limit values has been discussed in chapter 5.

SWELLING CHARACTERISTICS OF CLAY-SAND SYSTEM

A short review of the past investigations done on the swelling characteristics of clay has already been made in chapter 2. In predicting the swelling potential of clay, Seed⁷³ and others have established empirical formula for swelling potential in terms of clay content and activity of clay. Later on Ranganathan and Satyanarayana⁶³ suggested that shrinkage index should be used instead of plasticity index in predicting the swelling potential of clay. Holtz and Gibbs³⁶ also suggested the use of shrinkage limit value to predict the swelling behaviour of clay. The definition of activity according to Ranganathan and Satyanarayana⁶³ is the rate of change of shrinkage index with respect to change in clay content in the soil. Shrinkage index has already been defined as the difference between the liquid limit and shrinkage limit. The development of the empirical formula for swelling potential as derived in chapter 2 on the basis of experimental results is based on the assumptions that the clay content shrinkage index relation is linear and unique for a particular type of clay irrespective of the size and gradation of the inert component in the soil. So such a relation claims that the swelling potential of an expansive soil is the same whatever the gradation and nature of the inert component be so long as the type and amount of clay

is same. The sand taken for the above study was between B.S. 14 - B.S. 350. This covers a wide range of sizes. Still in nature fine sand clay mix or even silt clay mixes may be expected.

It was proposed to check the validity of such assumptions as mentioned above and the present investigation was made with a view to see whether size and gradation of sand has got any bearing on the swelling activities of an expansive mix.

4.1 Experimental investigation

For this purpose sands of various sizes were selected between sieve numbers:

B.S.	36 - 52	average size - 0.359 m.m.
B.S.	52 - 72	average size - 0.253 m.m.
B.S.	72 - 120	average size - 0.168 m.m.
B.S.	120 - 150	average size - 0.114 m.m.
B.S.	150 - 200	average size - 0.090 m.m.
B.S.	200 - 350	average size - 0.060 m.m.

Each of this sand was mixed with commercial Bentonite clay (obtained from Wyoming, U.S.A.) to various percentages of clay. The percentages of clay in the mixture were 5, 10, 20, 30, 40, 50 and 70. So for each grain size level sand, seven types of sand clay mixtures were obtained. The liquid limit and the shrinkage limit values were calculated for

each soil and the shrinkage index values were obtained and tabulated in table 4.1. A plot of clay percentage against shrinkage index values for all the six types of sand sizes are shown in figure 4.1.

4.2 Observations

(1) The shrinkage index varied significantly and linearly with respect to increase in clay content or decrease in sand content in the mixture.

(2) With decrease in the grain size of the sand in the mix, at any clay % level of the mix the shrinkage index does not deviate much but increases by a small amount.

(3) In figure 4.2 it was observed that the variation of shrinkage index with increase in grain size of inert material is nearly linear and the rate is low as it should be expected.

4.3 Discussion

From the observation of the shrinkage index-clay content curve it is obvious that shrinkage index maintains a linear relation with clay content but not at very low clay percentage. It can be stated that approximately up to 15% clay the relation is non-linear and after this the linearity stabilises. The linearity of the relation is not disturbed by the variation of the non-clay fraction which of course is slightly responsible for increasing or decreasing the slope of the shrinkage index clay content

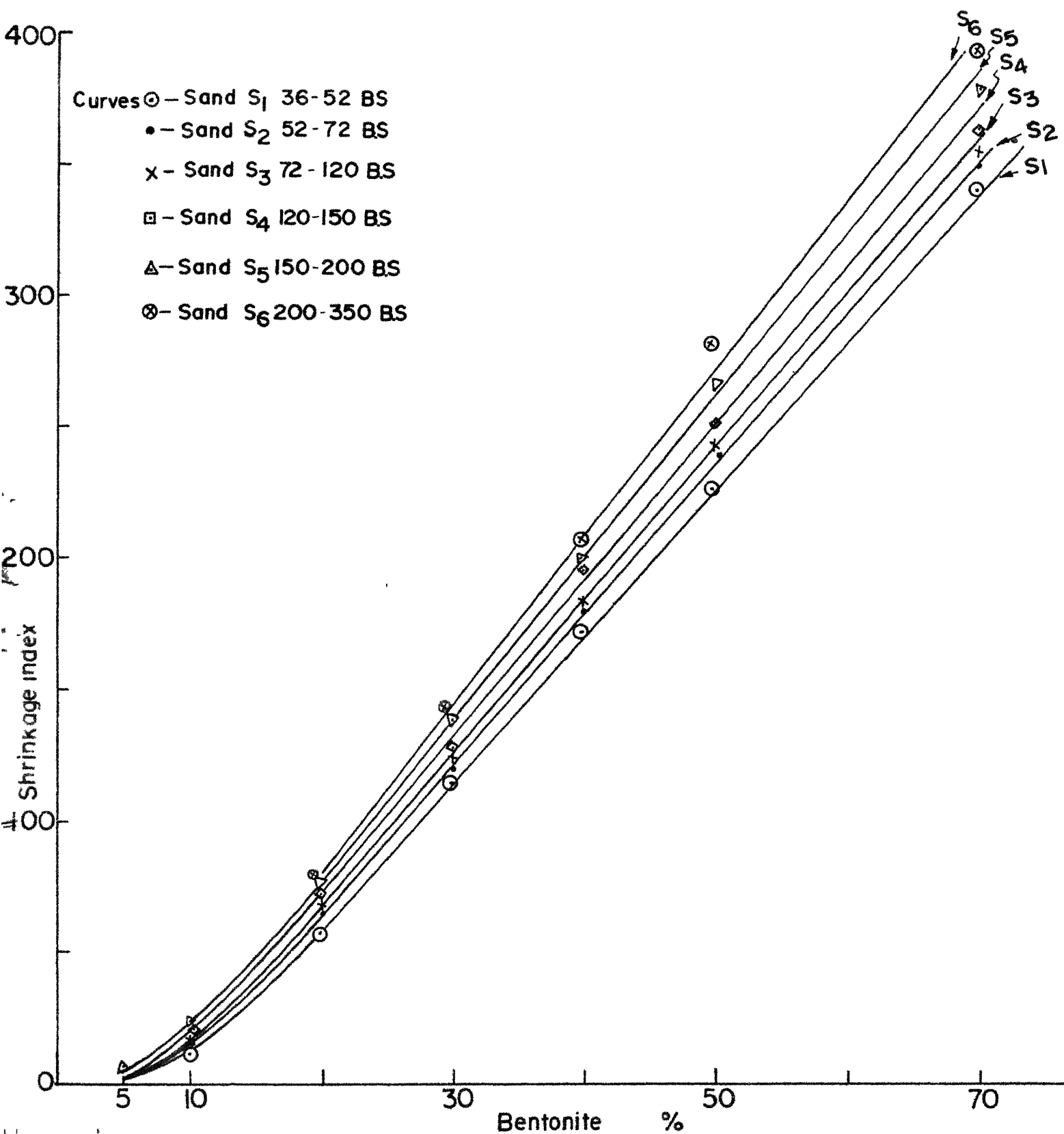


FIG. 4-1 VARIATION OF SHRINKAGE INDEX WITH CLAY PERCENTAGE FOR BENTONITE SAND SYSTEMS

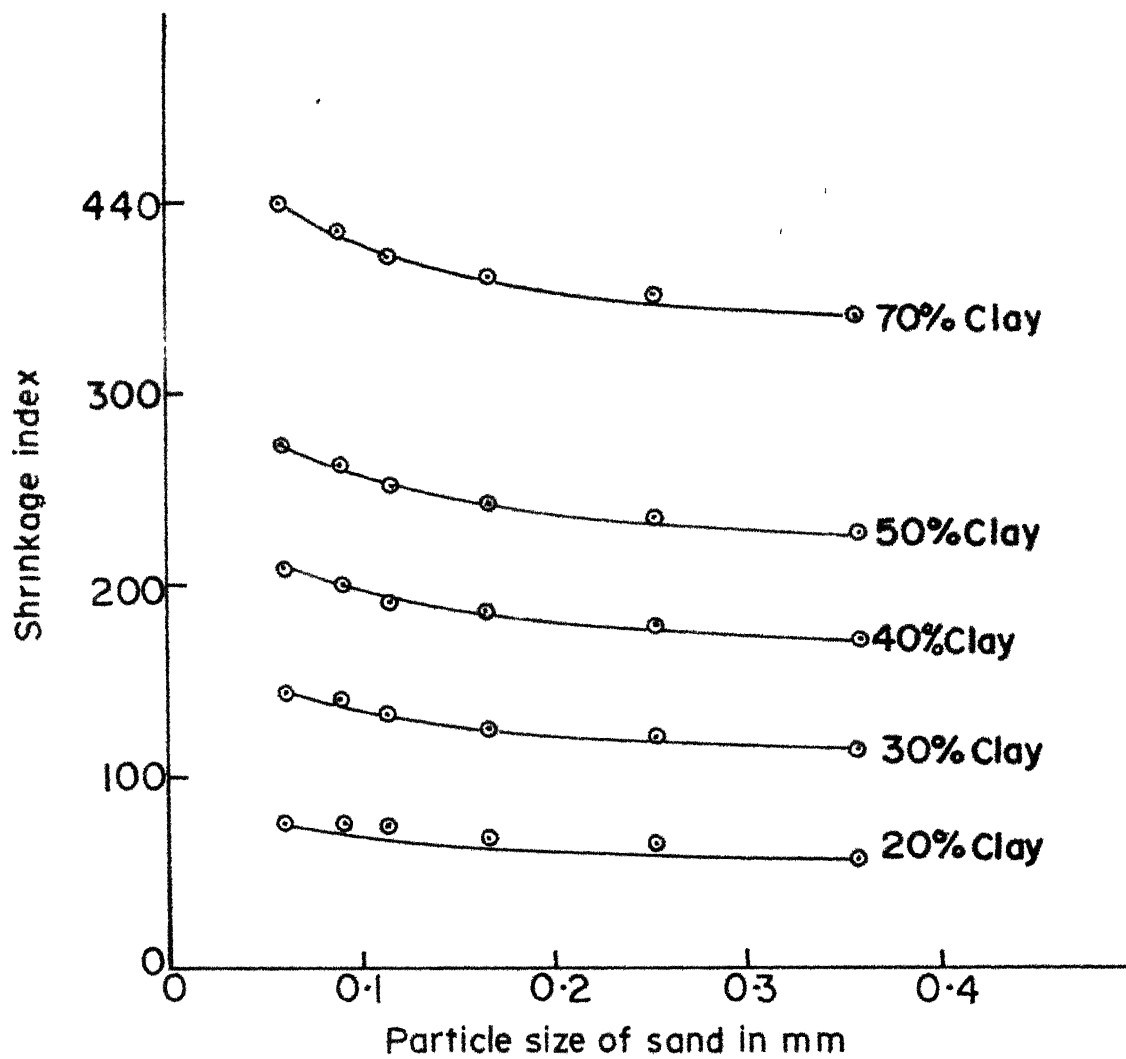


FIG 4.2 VARIATION OF SI WITH PARTICLE SIZE OF SAND FOR VARIOUS CLAY%

Table 4.1

Bentonite % in the mixture	Sand size in B.S. sieve																	
	B.S.		B.S.		B.S.		B.S.		B.S.		B.S.		B.S.		B.S.			
	36 - 52	L.L. S.L.	52 - 72	L.L. S.L.	72 - 120	L.L. S.L.	120 - 150	L.L. S.L.	150 - 200	L.L. S.L.	200 - 350	L.L. S.L.	350 - 425	L.L. S.L.	425 - 600	S.I.		
5	28	26	2	28.5	26	2.5	32	30	2	34.5	32	2.5	37	30	7	37	28	9
10	45	33	12	47	33	14	51	35	16	55	35	20	58	36	22	58	35	23
20	89	35.7	53.3	96	34	62	102	35.1	66.9	108.5	35	72.5	111	33.1	77.9	110	30.5	79.5
30	145.5	31	114.5	146	27	119	150.5	27.5	123	154	26.5	127.5	169	27.3	140.1	170	24.4	145.6
40	191	19	172	198	18	180	203	21	182	215.5	19	196.5	219	19.5	199.5	227	18	209
50	243	16	227	255	14	241	259	16.5	242.5	267	15.5	251.5	281	13	268	297	14.5	282.5
70	360	15	345	361	11	350	365.5	11	354.5	373	11	362	390	11	379	411	11.2	399.8

L.L.: Liquid Limit; S.L.: Shrinkage Limit; S.I. = L.L. - S.L.

line. So the activity of the soil is only slightly affected if the non clay fraction changes from very fine size to a coarse size.

This increase in the shrinkage index value of the soil with the decrease in sand size is in accordance with ⁴⁵ LeFevre's report on soil plasticity dependence on surface area. According to his report from the study of a variety of soils there is a linear correlation between specific surface area, liquid limit and plasticity index. In the present study when the sand size decreases the total surface area increases and hence there is an increase in liquid limit and consequently the shrinkage index, since the shrinkage limit is very small compared to liquid limit.

The average value of 'n' in the empirical formula ⁵⁹ derived for swelling potential by Satyanarayana was about 13. The 'n' has already been explained as the intercept on the clay content axis of the shrinkage index-clay content relation. The line corresponding to bentonite only showed a value of 'n' about 7 in the previous investigations. In the present study the value of 'n' for bentonite only is also found to be nearly 7 if the non-linear portion of the line below 15% of clay content is neglected.

A complete investigation of the above mentioned empirical formula for swelling potential taking the variation of sand size into account has not been pursued and deserves further study.

CHAPTER 5

SHRINKAGE CHARACTERISTICS OF CLAY-SAND SYSTEMS

The physico-chemical properties of multiphase mixtures cannot always be obtained by simply averaging of the properties of the individual phases. For such composite materials, the properties of the system are influenced by the state of existence of the phases involved. Studies in this direction for the properties of the various multiphase materials for industrial usage were reported by Franci and Kingery²⁴ (1954), Metzner⁵³ (1956), Meredith and Tobias⁵² (1962). Investigations have also been reported on mixing and packing in a multiphase system by Furnas²⁶ (1931), Oyama⁵⁹ (1939), Smith⁷⁶ (1929), Westman and Hugill⁸¹ (1930), White and Walton⁸² (1937), Hudson³⁷ (1949), Wise⁸⁵ (1952), Boerdijk¹⁰ (1952), Weidenbaum⁸⁰ (1958), Deresiewicz¹⁷ (1958), McGeary⁴⁹ (1961), Hogendijk³⁴ (1963) etc. In the present investigations, variations in the shrinkage limits of various clay-sand mixes have been investigated and explained in terms of state of existence of these two participants.

5.1 Experimental investigations

The properties of the bentonite, kaolinite and sand used in the present investigation are shown in table 5.1.

Table 5.1

Properties	Bentonite	Kaolinite	Sand
L.L.	640	51	
P.L.	60	30	
S.L.	10	23	
Sp. Gr.	2.3	2.52	2.65

The sand has been fractionated into six grain size groups: 36-52, 52-72, 120-150, 150-200 and 200-350 mesh ranges on the B.S. sieve. With each of these sand, mixtures of bentonite-sand, kaolinite-sand have been prepared with varying percentages of clays as indicated in table 5.2 (for bentonite) and 5.3 (for kaolinite). Shrinkage limit for all these mixes at each of the above grain size levels of sand have been determined in the laboratory adopting conventional procedure (A.S.T.M. standards, 1958). The results are indicated in table 5.2 and 5.3 and the values of shrinkage limit are plotted against clay percentage in figs. 5.1 and 5.2.

5.2 Results

Bentonite-sand system

At any grain size level of sand, the trend of variation in the shrinkage limit values for bentonite-sand mixes is as follows:

1) With increase in percentage of bentonite, shrinkage limit initially builds up and attains a maximum value (fig. 5.1, table 5.2).

2) After the maximum value of shrinkage limit is attained, any further increase in the bentonite percentage brings down the shrinkage limit value at a relatively rapid rate.

BENTONITE SAND SYSTEMS

BENTONITE SAND SYSTEMS WITH PARTICIPATING SAND SIZES

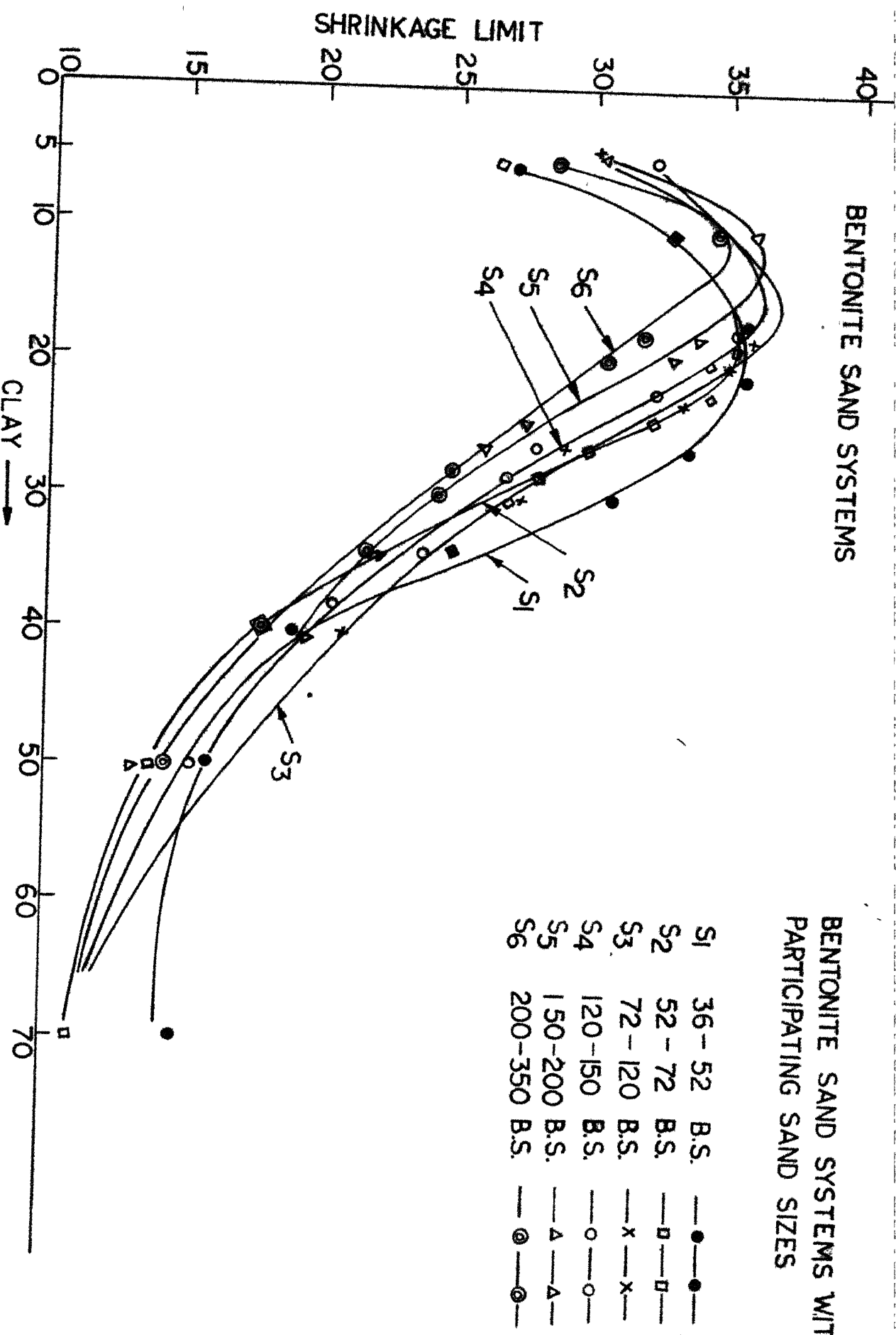


FIG. 5.1 SHRINKAGE LIMIT CLAY PERCENTAGE RELATION FOR MIXTURES OF BENTONITE AND SAND

Table 5.2

Bentonite % in the mixture	Shrinkage limit values for different sand sizes					
	36 - 52 B.S.	52 - 72 B.S.	72 - 120 B.S.	120 - 150 B.S.	150 - 200 B.S.	200 - 350 B.S.
5	26	26	30	32	30	28
10	33	33.2	35	35	36	35
18	35.4	35.6	36	35.4	34	32.1
20	35.7	34	35.1	35	33.1	30.5
22	35.5	34.3	33.3	32.5	29.1	29.2
24	34.7	32.2	32.1	28.2	27.5	28.6
26	33.7	30	29.1	30.8	26	28.3
28	32	28.1	28	26.7	26.1	25
30	31	27	27.5	26.5	27.3	24.4
34	25.3	24.8	25.1	24	22.3	21.8
38	20.8	20	20	20.4	19.9	18.5
40	19	18	21	19	19.5	18
50	16	14	16.5	15.5	13	14.5
70	15	11	11	11	11	11.2

KAOLINITE SAND SYSTEM, PARTICIPATING SAND SIZES:

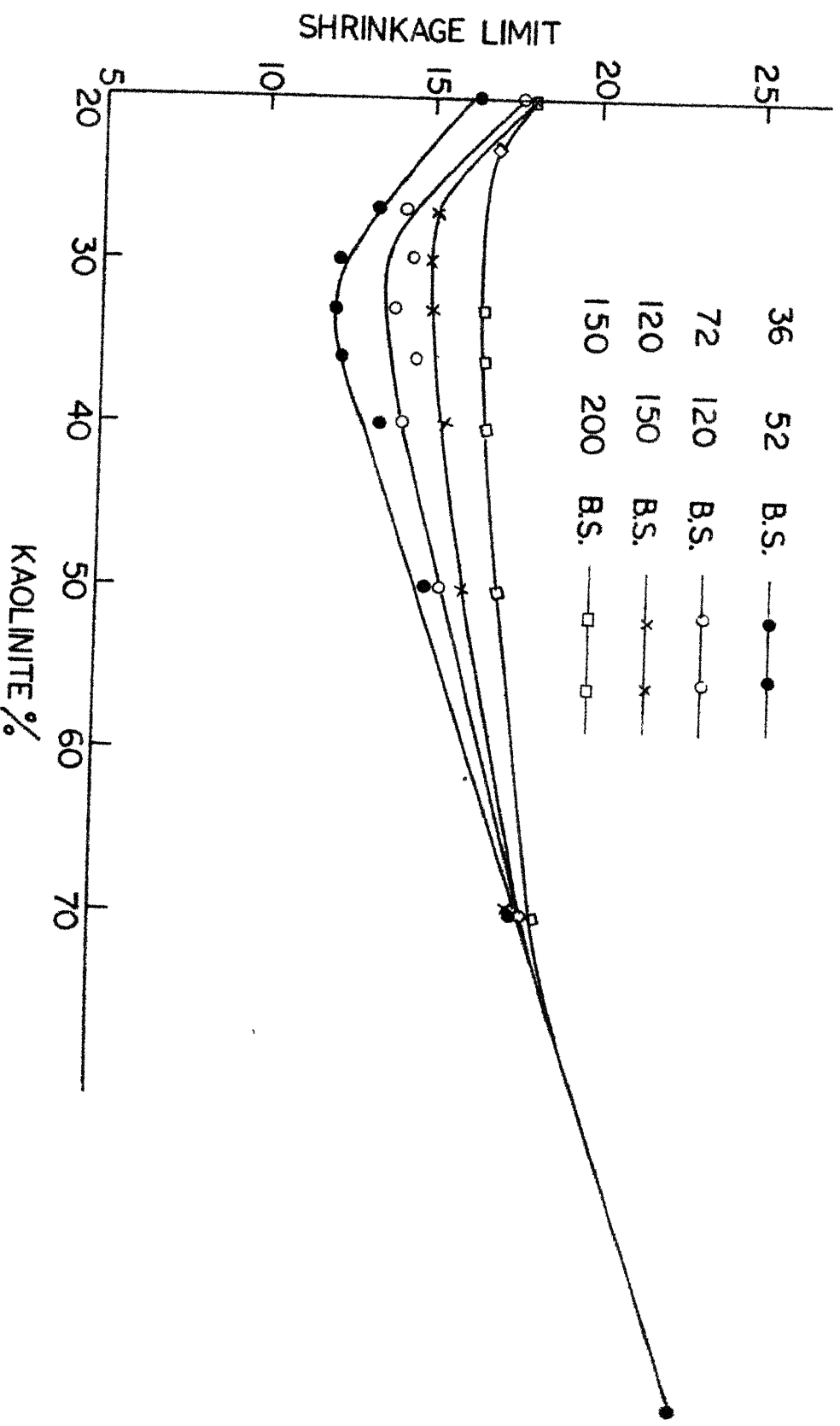


FIG.5.2

Table 5.3

Kaolinite % in the mixture	Shrinkage limit values for different sand sizes			
	36-52	72-120	120-150	150-200
20	16.3	17.6	17.9	18
23				16.9
27	13.3	14.1	15	
30	12.1	14.5	14.9	
33	12	13.8	15	16.5
36	12.2	14.5		16.6
40	13.5	14.1	15.5	16.8
50	15	15.2	16.05	17.2
70	17.6	17.9	17.6	18.3

3) At higher levels of clay participation the shrinkage limit curve flattens, with a tendency for the values to approach that for pure bentonite (fig. 5.1).

4) With finer grain sizes of participating sand in the mix, the maximum value of shrinkage limit for the system is attained earlier (with comparatively lesser amount of participating clay).

Kaolinite-sand system

However with kaolinite instead of bentonite the trend of changes in the system as evidenced from fig. 5.2 are somewhat different and as follows:

1) An initial and gradual decrease in shrinkage limit for the system with increasing kaolinite participation up to a certain limit (depending on the grain size of sand in the mix).

2) The shrinkage limit attains a minimum value at certain clay percentage.

3) Any subsequent increment in participating clay builds up the shrinkage limit until it approaches the value of that of pure kaolinite.

4) It can also be seen from fig. 5.2 and table 5.3 that the minimum value of shrinkage limit attained for any clay-sand mix depends also on the grain size of the participating sand. With finer grain sizes of sand the minimum value of shrinkage limit is attained earlier and shape of the variation curve is also modified.

5) Where as the peak values of the shrinkage limit for bentonite-sand systems for different grain sizes of sand are attained with bentonite varying in amount 12% - 20% (fig. 5.1), for kaolinite-sand systems the corresponding critical values of shrinkage limit are attained between 23% - 35% (fig. 5.2).

5.3 Discussion

The clay-sand system is a heterogeneous one and can be approximated to a binary mix with two grain-size phases participating. One of the components is extremely small in size (of colloidal clay) and the other is coarser (sand) size. The state of packing and theoretical critical density distribution for such a binary system of spheres has been reported by ⁴⁹McGeary (1961). As already stated in Chapter 3, ⁴⁹McGeary's studies involved packing induced by mechanical means. Both the constituents in the binary system in his investigation were of the same composition and of inert material. As such the present study differs from ⁴⁹McGeary's work from the following aspects:

- 1) The present system involves multiphase components with differing grain sizes.
- 2) One of the components (bentonite) is active in presence of water in case of bentonite-sand systems.
- 3) The state of packing is induced naturally during dehydration and shrinkage of the clay-sand system with water.

4) The specific gravities of the two different components along with the percentage levels of the participants would control the bulk density of the mixture. While the critical packing and maximum density of the system coincide in a homogeneous binary system (such as the one studied by McGeary⁴⁹) it need not be so in the case of a heterogeneous system (with two phases of differing compositions and specific gravities).

It has already been discussed in Chapter 2, that kaolinite because of its electrostatic neutrality is inert in its relation to water while montmorillonite (a predominant constituent of bentonite) adsorbs water into its lattice due to weaker bonding in its interlayered space as also due to charge deficiency. As such, during shrinkage studies of these materials with sand in presence of water, it is useful and necessary to conceive the behaviour of clay sand-systems as follows:

I. Systems with one component active and the other inert
(Inert-active system)

The moisture that is involved in the dehydration would be not only pore water but a substantial amount also present as lattice water in montmorillonite. As such the system behaviour would depend on the dehydration conditions existing in the system. Since the active component shrinks considerably during dehydration, there is a tendency for the system surface to be hard and consequent isolation of the trapped active component in the available pore spaces of the coarser inert

component at lesser clay levels, thus retarding dehydration to certain extent. However if the active component dominates in the system so as to be in continuity at every place within the system as also through the system surface with the outside, rapid dehydration is possible with the active component loosing considerable moisture. Consequently shrinkage would also be very prominent in the mix. The extreme example of such a case is either a montmorillonite-sand mix with very minor sand content or a pure montmorillonite clay. In both these cases, the shrinkage is considerable and shrinkage limit is quite low.

II. Binary systems with both components inert (inert-inert system)

In this system, since both the components are inert in presence of water, the moisture that is involved in the dehydration of the system would be mainly the pore water in the voids with a thin film around individual particles. In such a case, if the voids of sand are gradually filled with increasing clay, the available pore space for moisture decreases and under the densest critical packing in the clay-sand system, the moisture at shrinkage limit would be the least.

With the above models, the trend of variation of the shrinkage limit values for any clay-sand mix can be explained as follows:

(a) Bentonite-sand system

This is equivalent to a heterogeneous system having one component active and the other inert. In such a system with

low clay percentage in the clay-sand mix, the clay would occupy the voids created by the packing of the coarser sand component, when the coarser sand particles are drawn together into physical contact during dehydration and shrinkage. In such a situation, the sand particles would be in a state of continuity while the clay particles in the void remain in a discontinuous state. Consequently the clay in the system has only a minor influence over the shrinkage limit of the system. Dehydration of the trapped clay would also be dependent on the environmental condition of the interior of the system whose surface is almost sealed off due to the complete dehydration of bentonite and consequent hardening of the surface of the system. Further addition of bentonite to the system, so long as clay continues to fill the voids during shrinkage process in the system, would contribute to the building up of shrinkage limit.

A critical packing condition would occur when all the sand voids are filled to the maximum by the participating clay particles. Such a situation corresponds to inflexion points in the curves shown in fig. 5.1. Any further addition of clay however would bring drastic changes in the property of the system. During shrinkage, as the particles are drawn together, the finer clay particles in addition to filling the voids also occupy the intergranular space of the sand grains. As such the clay particles are no longer in a state of discontinuity and greater dehydration of the bentonite in a bentonite-sand

system is then possible. All the clay particles would be in a state of continuity with each other and with the system surface. The system under such circumstances would be influenced to a major extent by the participating clay. The shrinkage limit would now be lower due to the physical continuity and consequent rapid dehydration of bentonite. With further addition of clay to the mix, the system would have a tendency to rapidly attain the shrinkage limit of pure bentonite as shown in figure 5.1.

(b) Kaolinite-sand system

This system corresponds to the inert-inert system described in the preceding paragraphs. At lower percentage levels of kaolinite in the kaolinite-sand mixes, the kaolinite occupies the voids created by packing of the sand particles and the sand particles are in continuity. Since the pore water is the main moisture, the shrinkage limit for different mixes with increasing kaolinite in these sand voids would be correspondingly lower with dehydration. With continued addition of kaolinite into the system, a stage would arrive when the kaolinite practically fills all the sand voids and the shrinkage limit in such a critical situation would naturally be the lowest. Any subsequent addition of kaolinite would destroy the continuity of the sand particles in the system and the clay attains continuity. As such the system is more influenced by the nature of clay fraction present and the shrinkage limit variation from the critical state would be tending to the value for pure kaolinite.

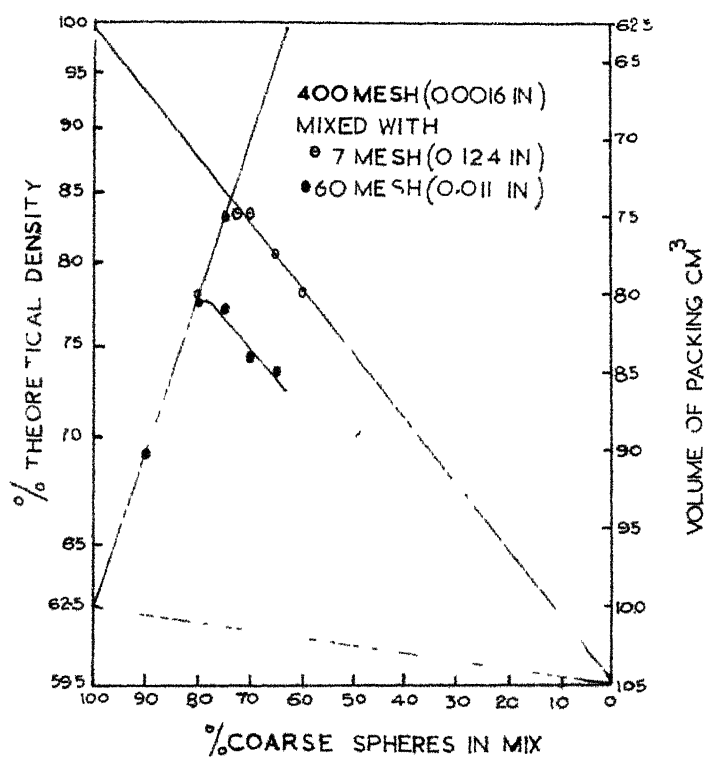


FIG. 5-3. BINARY MECHANICAL PACKING OF COARSE SPHERES WITH 400 MESH TUNGSTEN SPHERES (AFTER McGEARY, 1961)

As such the curve indicating the trend of variation of shrinkage limit for any kaolinite sand mix (fig. 5.2) would generally follow a decrease or downward trend up to the critical packing situation and an increasing trend then onwards.

5.4 Influence of grain size of participating sand in the clay-sand mixes

In a binary system, it has been theoretically and experimentally shown by earlier investigators that the maximum packing density that can be achieved is a function of the ratio of the participating grain sizes of the two phases. The critical packing condition (where all the available voids created by the packing of the coarser particles are filled in with the finer participants) is attained earlier with the decreasing ratio of coarse to fine size. Fig. 5.3 indicates such a situation of the dependence of critical packing in a binary system on the participating grain sizes as verified by McGeary.⁴⁹

In the present investigation, as can be seen from figs. 5.1 and 5.2, the critical packing condition indicated by the inflexion points of the shrinkage limit curves occurs with lesser amount of clay in the clay-sand mixes for finer sizes of sand participating. That is, with increasing fineness of the participating sand in the clay-sand mix, the critical packing occurs with a lower clay content.

It is also interesting to note from the present investigations that while the critical packing condition is seen to exist with clay content between 23 to 35% in case of kaolinite-sand system depending on the grain size of sand, in case of bentonite sand system for the corresponding grain sizes of the participating sand, the critical packing exists in the region of 12 to 20% of clay. This can be explained by the fact that bentonite with water swells enormously and as such occupies greater volume than equal amount of kaolinite under identical moisture conditions.

CHAPTER 6

X-RAY INVESTIGATION OF SHRINKAGE PROCESS IN BENTONITE

X-ray diffraction method has been extensively applied to study the crystalline structure of clay minerals. It has already been discussed that clays constitute of layered structures and the surfaces of the layers are electrically unbalanced for which water molecules are adsorbed into the spaces between each layer depending on the type of clay and the amount of charge deficiency, causing hydration and volume increase in the clay. The increase in lattice spacing in C axis direction due to adsorption of water in steps of unit layers has been successfully verified and calculated with the help of x-ray diffraction technique by Bradley, Grim and Clark⁸ and many other investigators.

During the process of dehydration these adsorbed water molecules escape from the clay structure again the number of layers going out being integral. Obviously the number of unit layers of water escaping depends upon the period of drying, temperature to which the wet clay sample is exposed and the relative humidity. The present study attempts to investigate the effect of time on the dehydration of clay sand mixes which are subjected to almost identical temperature and humidity conditions.

X-rays are electromagnetic radiations of very small wave lengths of the order of 0.01 to 100 Å and the phenomenon of diffraction involves the scattering of x-rays by atoms of a crystal in definite directions away from the crystal. The direction of the scattered rays is quantitatively related to the distance of separation of atomic planes by Bragg's law; $n\lambda = 2d \sin\theta$ where n is the order of reflection, λ is the wave length, θ is the reflection angle and d is the interplanar spacing.

Each crystalline substance has its own characteristic atomic structure which diffracts x-rays in a characteristic pattern. The analysis of the pattern establishes uniquely the diffracting substance. The x-ray diffraction method is non-destructive and needs a very small amount of material. It can be used for quantitative analysis of mixtures. Detailed information on the application of X-ray diffraction technique to study the crystalline clay structure and the underlying principles involved in the method have been given by, Rich & Kunze⁶⁵, (1964), Brindley¹¹ (1951), MacEwan⁴⁷ (1951), George²⁷ (1961), Nagelschmidt⁵⁷ (1936), Black, Evans, Ensminger, White and Clark⁷, Barshad⁵ (1949) and Norrish⁵⁸ (1954)



G. E. C. X-RAY DIFFRACTOMETER

6.1 Instrumental set-up

X-rays are produced when high speed electrons collide on a metal target. The x-ray tubes contain two electrodes, an anode (the metal target) maintained with few exceptions, at ground potential, and a cathode maintained at high negative potential. Vacuum is maintained in the tube. The cathode is a tungsten filament and the anode is a water cooled block of copper containing the desired target metal at the end. The cathode is connected to a transformer when electrons emit and then collide on the anode (target) which in turn emits x-rays which pass through windows with screen such that undesired $k\beta$ radiations are screened and $k\alpha$ radiations pass out. The target is water cooled to prevent melting.

The x-ray produced is made to fall on the sample mounted on a diffractometer which is made to rotate through desired an angle. The diffracted x-ray then enters into a movable counter and amplifier. The output is fed into an automatic recorder with a chart paper on which the diffraction pattern of the sample is recorded. The angle of rotation is read on the graduated scale of the diffractometer. Further details about x-ray diffractometer and technique have been explained by Cullity¹⁵ (1956), McLachlan⁵¹ (1966) and Bacon⁴ (1957).

In the present investigation a General Electric x-ray diffractometer with cobalt and chromium targets was used. A chart paper speed of $2^\circ/\text{inch}$ and a motor speed of $2^\circ/\text{min.}$ for

the diffractometer were utilised. Reflections scanned with 500 c.p.s. and 1000 c.p.s. were in the range $18^\circ - 35^\circ$ with Co $k\alpha$ and $6^\circ - 60^\circ$ with Cr $k\alpha$.

6.2 Experimental investigations

The clay sand mixes chosen for the present study consist of very fine sand from 200 - 350 B.S. sieve mixed with 20%, 50%, 70% of clay by weight of the total mixture. The samples were saturated with the same water content at which corresponding shrinkage limit tests were performed as discussed in the previous chapter. All the samples were put to identical temperature and humidity conditions. Samples of pure bentonite and the same with different percentages of sand at various stages of dehydration process for different time periods were analysed by standard x-ray diffraction procedure. The x-ray diffraction patterns were analysed to investigate the variation of lattice spacing for certain reflections of clay during the process of dehydration.

6.3 Results and discussion

The diffraction patterns for various planes are shown in fig. (6.4). The reflections selected for the purpose of comparison were:

- (1) (001) reflection corresponding to 12.44 Å lattice spacing in pure montmorillonite.

Table 6.1

Sample	Radi- ation	Fresh moist 2 θ d(A°)	Dehydration period										
			12 hours 2 θ d(A°)	24 hours 2 θ d(A°)	36 hours 2 θ d(A°)	48 hours 2 θ d(A°)	72 hours 2 θ d(A°)						
Pure bentonite	Cr K α	8.8	14.4	8.23	15.93	8.13	16.17	10.28	12.78	10.47	12.53	10.67	12.32
	Cr K α	29.49	4.5	29.38	4.51	29.38	4.51	29.38	4.51	29.38	4.51	29.57	4.49
	Cr K α	39.8	3.36	39.71	3.37	39.64	3.38	39.87	3.36	39.88	3.36	39.89	3.35
20% bentonite+ 80% sand	Cr K α	9.8	13.4	10.11	12.98	10.27	12.78	10.27	12.78	10.29	12.75	10.72	12.25
	Co K α	32.03	3.22	31.7	3.27			31.93	3.25			32.0	3.24
50% bentonite+ 50% sand	Cr K α	9.43	13.34	8.5	15.44	10.30	12.75			10.45	12.67	10.45	12.67
	Cr K α	29.4	4.51	29.4	4.51	29.46	4.5			29.41	4.5	29.41	4.5
	Co K α	31.04	3.34			30.81	3.37			30.85	3.36	30.95	3.35
70% bentonite+ 30% sand	Co K α	24.15	4.28			24.22	4.26			24.33	4.24	24.38	4.23
	Co K α	30.95	3.35			31.0	3.35			31.07	3.34	31.05	3.35

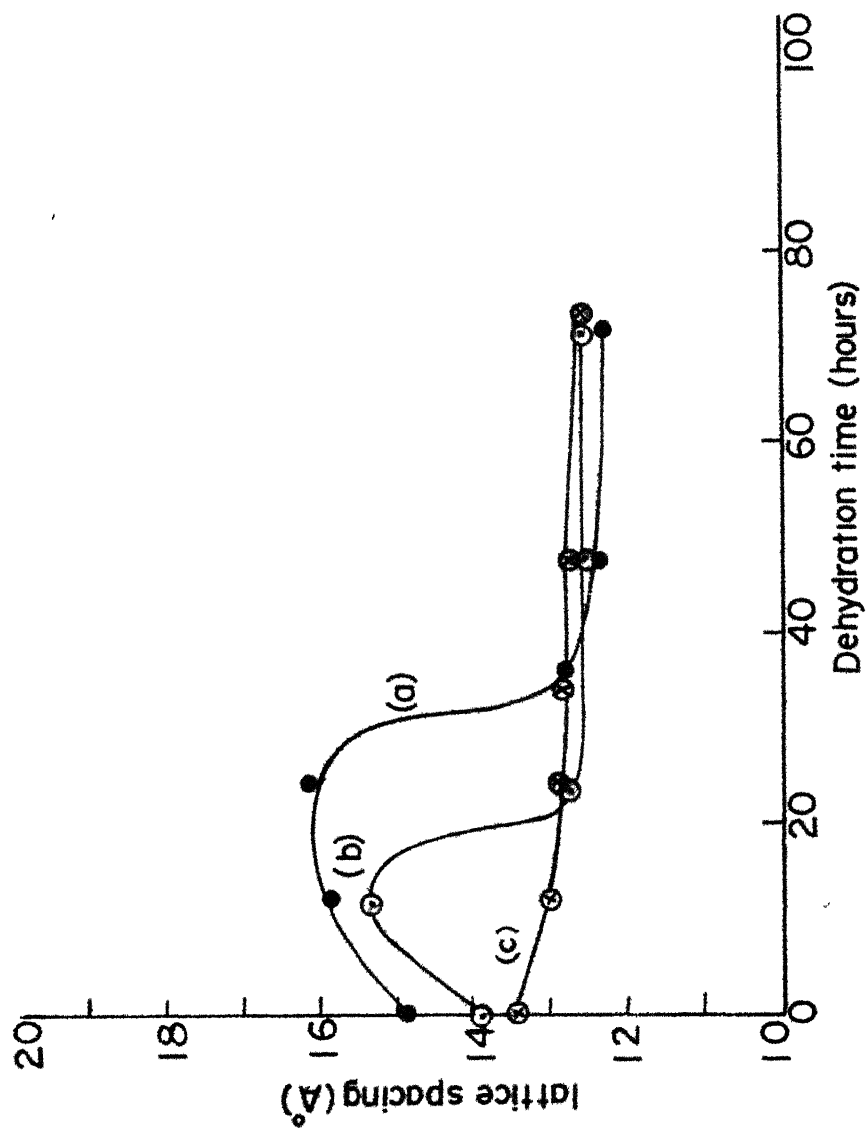


FIG. 6.1 LATTICE SPACING VARIATION FOR 001 LINE OF MONTMORILLONITE WITH TIME DURING DEHYDRATION IN (a) PURE BENTONITE ($\text{Cr K}\alpha$) (b) 50% BENTONITE + 50% SAND ($\text{Cr K}\alpha$) (c) 20% BENTONITE + 80% SAND ($\text{Cr K}\alpha$)

(2) Reflections corresponding to lattice spacings 4.48 \AA° and 3.35 \AA° . The variations of lattice spacing with dehydration time in hour is given in table. 6.1 and plotted in figures 6.1, 6.2 and 6.3 for pure montmorillonite and montmorillonite with sand.

The present investigation involves the study of the dehydration process of montmorillonite by x-ray diffraction method with randomly oriented samples. Consequently the patterns for several reflections indicate a gradual dehydration even though dehydration in reality is step-wise. Such gradual decrease in lattice is noticed in both pure montmorillonite and the different montmorillonite sand mixes in figures 6.1, 6.2 and 6.3 showing that dehydration is a continuous process.

It has been observed earlier by Bradley, Clark and Grum⁸ for Wyoming bentonite that the swelling of the lattice during hydration took place in a step-wise fashion. A series of four apparently definite and discrete hydrates were found, having cell heights of 12.4, 15.4, 18.4 and 21.4 \AA° . Near the range where any given hydrate is stable, successive orders of (001) reflections appear for it alone. In intermediate ranges two suits of (001) reflections appear simultaneously, one to be identified with the hydrate next higher in the sequence one with the hydrate next lower.

In their study they used oriented aggregates in which the basal cleavages were substantially parallel. Hofmann, working with powders, failed to check their conclusions, but later Hofmann and Housdorf,⁴³ using oriented aggregates, also concluded that the hydration of montmorillonite took place by the formation of successive monomolecular layers of water. The latter investigators and later Hendricks and Jefferson³² have shown theoretically that powder diffraction should indicate that the c-axis-dimension spacing varies continuously but not uniformly with water content, and this apparently would be a result of an averaging effect from a lattice that contains various numbers of water layers in different parts. That is, the apparently continuous change in c dimension results from a random alternation of successive discrete hydrates.

Fripjat, Chaussidon and Tauillaux²⁵ have studied the dehydration phenomena of montmorillonite by infrared spectroscopy and have also shown that the dehydration process of montmorillonite observed in a random sample is continuous despite the stepwise hydration with discrete water molecules being adsorbed. According to Fripjat, Chaussidon and Tauillaux,²⁵ during dehydration one reflection only, characteristic of interstratification, can be observed. In the present investigation the (001) reflection corresponds to 12.44 Å° which expands in c dimension after saturation and its decrease with dehydration time is noticed to be continuous

though not stepwise. This is because of the random orientation of the particles in the clay in the sample tested. Also Roderick⁶⁸ and Senich⁷⁴ (1966) have investigated on the hydration and dehydration of montmorillonite leached with sodium and calcium chlorides. According to their reports the observed first order basal spacing changes in a continuous but non-uniform manner; continuity is due to simultaneous existence of varying number of molecular layer of interlayer water. The expansion occurs in three increments; prior to each increment, most clay plate-lets are at one separation corresponding to an integral number of molecular layers of water. The relation between relative humidity and basal spacing depends on the source and method of preparation of sample, the initial condition of the sample and whether data is collected during adsorption or desorption.

The same report says during desorption the 3\AA° and 5\AA° regions contained the most intense secondary reflections which were explained on the basis of layer electron density distribution in the clay structure. The present set of records reflect such type of intense peaks in the same region and this can be concluded as a positive behaviour of the montmorillonite clays.

From the figure 6.1 it is observed that in the initial stages swelling in moist montmorillonite stabilises in the first 24 hours after which the actual dehydration process starts. It is also noticed that the rate of dehydration seems to be high during initial stages and the curve flattens out towards the end. Such behaviour is pronounced also with montmorillonite sand mixtures.

However, the rate of dehydration for montmorillonite with sand is different from that in pure montmorillonite depending on the percentage of sand in the mixture. With higher sand content the rate of dehydration is lower as can be clearly seen in figure.

From fig. 6.2 and 6.3 it is observed that for other reflections also the corresponding nature of dehydration is similar but the rate of change of lattice spacing with time is very small in both pure montmorillonite and montmorillonite mixed with sand. The changes are not as conspicuous as with (001) reflections which indicates to the anisotropic shrinkage behaviour of montmorillonite. The shrinkage or swelling is maximum in c direction.

6.4 Summary

The conclusions from x-ray study can be summarised as:

- (1) It takes about 24 hours for moist montmorillonite to stabilise and in this period the lattice is seen to be under adjustment during swelling.

(2) During dehydration the lattice water (inter layer water) in the montmorillonite participates.

(3) The swelling and shrinkage of montmorillonite is anisotropic, the maximum being in c direction (perpendicular to 001 plane).

(4) Even though the water molecules adsorbed are in integral number, no discrete or stepwise variation of lattice spacing during shrinkage is noticed. The changes in lattice spacing in montmorillonite are gradual due to random orientation of the sample and differing states of hydration of different planes.

(5) The rates of variations of lattice spacing during dehydration for various planes for pure montmorillonite is different from that in montmorillonite along with sand indicating the influence of sand on the dehydration rate of montmorillonite.

Before actual dehydration starts the magnitude of the (001) basal spacing in c direction for pure montmorillonite is higher than that in a montmorillonite sand mixture.

CHAPTER 7

SYNTHESIS

The present investigations were mainly concerned with the influence of clay composition, percentage of the participating clay as also of the grain size of the sand phase in heterogeneous clay-sand systems on the swelling and shrinkage behaviour of the system. As stated in the chapters 3 and 4, not much has been done by the earlier workers on these aspects. In nature, any soil can be approximated to a heterogenous system with granular and colloidal phases. It can be a binary system (involving only two phases) or it can also be a ternary or quaternary system depending upon the grain sizes of the phases involved. As such in understanding the engineering behaviour of a soil, a basic understanding of the role and participation of the individual phases or constituents in the multiphase system, soil, is necessary and relevant. With that view the investigations that have been conducted comprise the present work and the basic conclusions that were arrived at on the basis of such a study are as follows:

- 1) Except at a very low percentage of the participating clay, the shrinkage index of the system has a significant linear variation with clay (bentonite) content in a clay-sand system.
- 2) The finer the grain size of the inert component (sand) in the system, the greater is the slope of the curve of variation of ~~shrinkage~~ shrinkage index with clay percentage.

- 3) The variation of shrinkage limit in a clay-sand system depends upon the state of packing of the individual grains. The variation takes place at two rates - one up to the critical packing condition (until which the clay particles continue to occupy the sand voids) and the other beyond that condition wherein the clay particles attain a continuity and influence predominantly the system.
- 4) In case of kaolinite sand system, as expected in an inert-inert binary system, since the moisture mainly occupies the pore spaces, the shrinkage limit falls down with increasing clay content in the system until the critical packing condition where it touches a minimum value. Beyond this stage, further additions of clay brings in continuity all the clay fraction and the system starts building up the shrinkage limit with a tendency to reach the pure clay value.
- 5) However, in the bentonite-sand systems, a drastic deviation to this general behaviour is noticed due to active nature of bentonite. In bentonite considerable moisture is taken in the interlayered space in the lattice and as such dehydration depends upon the state of existence of bentonite particles. Up to the critical packing condition, the clay particles are isolated and the system surface is hardened and the dehydration

is retarded with a consequent increase in the shrinkage limit reaching a maximum at the critical packing condition. Any subsequent addition of clay would bring down the shrinkage limit since the clay particles attain a continuity within themselves and with the system surface. Rapid dehydration is possible under such circumstances.

- 6) For any particular grain size of participating sand in the clay-sand-system, with bentonite the critical shrinkage value corresponding to the critical packing condition is reached earlier (at lower participating clay percentage) than with kaolinite in the system. This can be easily understood since bentonite with water occupies a greater volume than equal percentage (by weight) of kaolinite with water and consequently the sand voids can be filled up quicker with bentonite than with the same amount of kaolinite.
- 7) With increasing fineness of the participating sand in a clay-sand system, it has been found that the critical packing condition corresponding to the critical shrinkage limit value reaches earlier (with lesser clay content).
- 8) X-ray studies have indicated that when a bentonite-sand system is prepared at certain moisture condition, usually it takes about 24 - 26 hours for the bentonite to stabilize in swelling till which time the lattice expands. Shrinkage in lattice is seen only after that period in case of bentonite.

- 9) It was also observed from the x-ray diffraction studies, that the rate of dehydration of montmorillonite (the dominant constituent of bentonite) is influenced by the sand fraction that is present in the system and that the lattice water participates in the dehydration process.
- 10) The changes in the lattice for montmorillonite either during hydration or dehydration are pronounced for the 001 planes (in c-direction) than in any other direction indicating the anisotropic behaviour of montmorillonite swelling and shrinkage.
- 11) For unoriented samples, it was observed for hydration or dehydration, a gradual increase or decrease respectively of the lattice is noticed. Though hydration or dehydration is discrete and stepwise (with integral number of water molecules involved), due to random orientation of the sample a gradual change is recorded in x-ray patterns. This has also been observed by earlier workers as stated in the preceding chapters.

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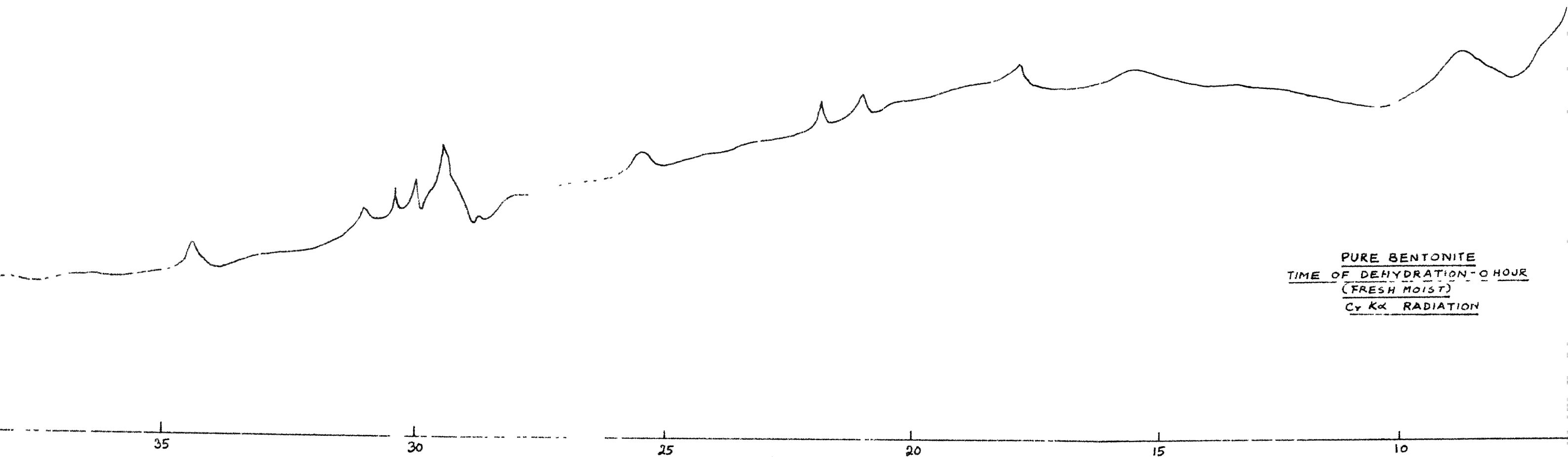
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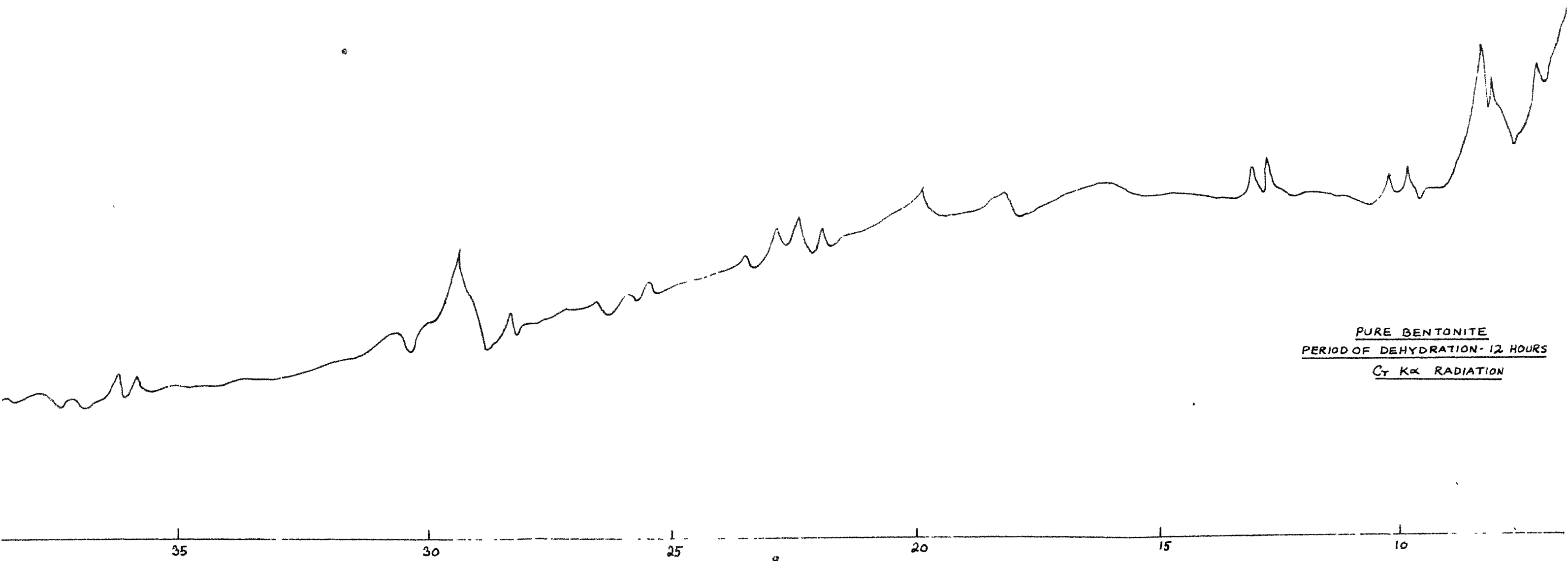
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APPENDIX
X-RAY RESULTS



PURE BENTONITE
TIME OF DEHYDRATION - 0 HOUR
(FRESH MOIST)
Cy K α RADIATION

2 θ
Fig 6 4-1



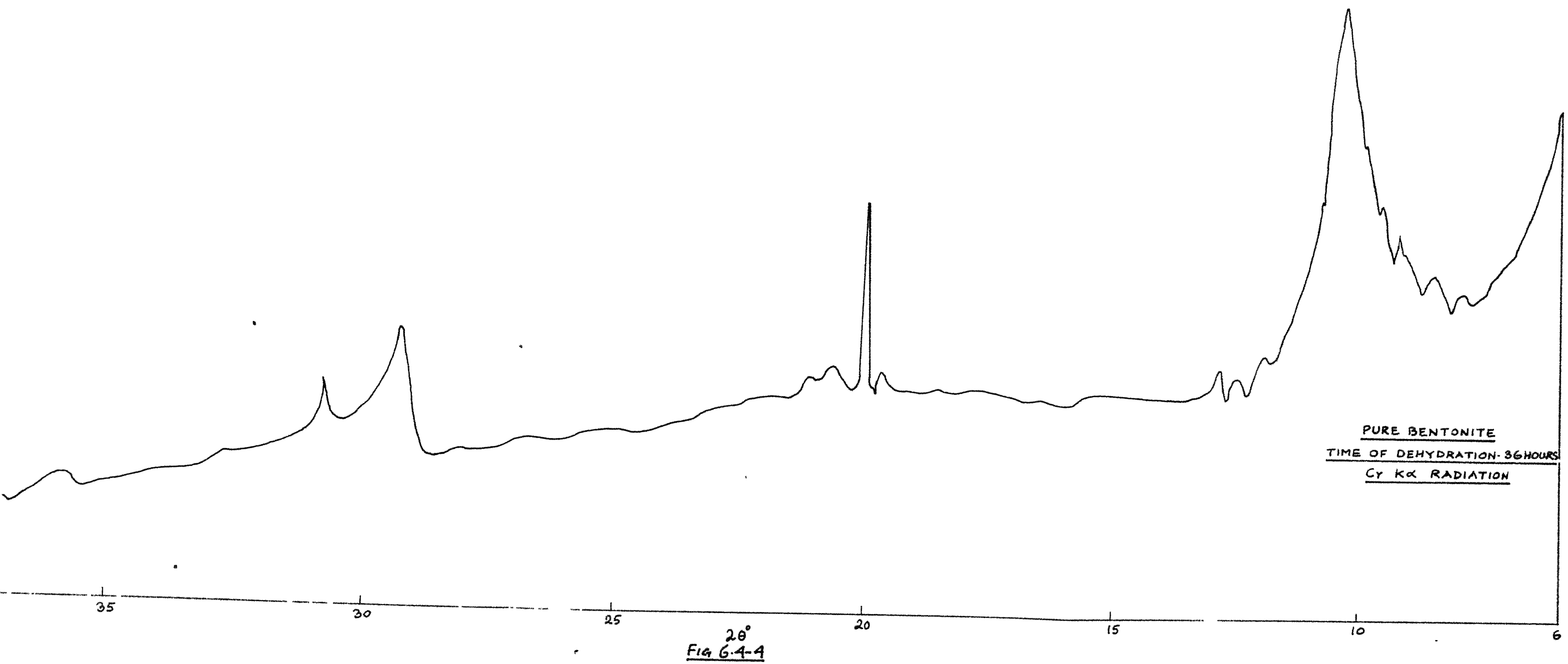
PURE BENTONITE
PERIOD OF DEHYDRATION - 12 HOURS
C_T K α RADIATION

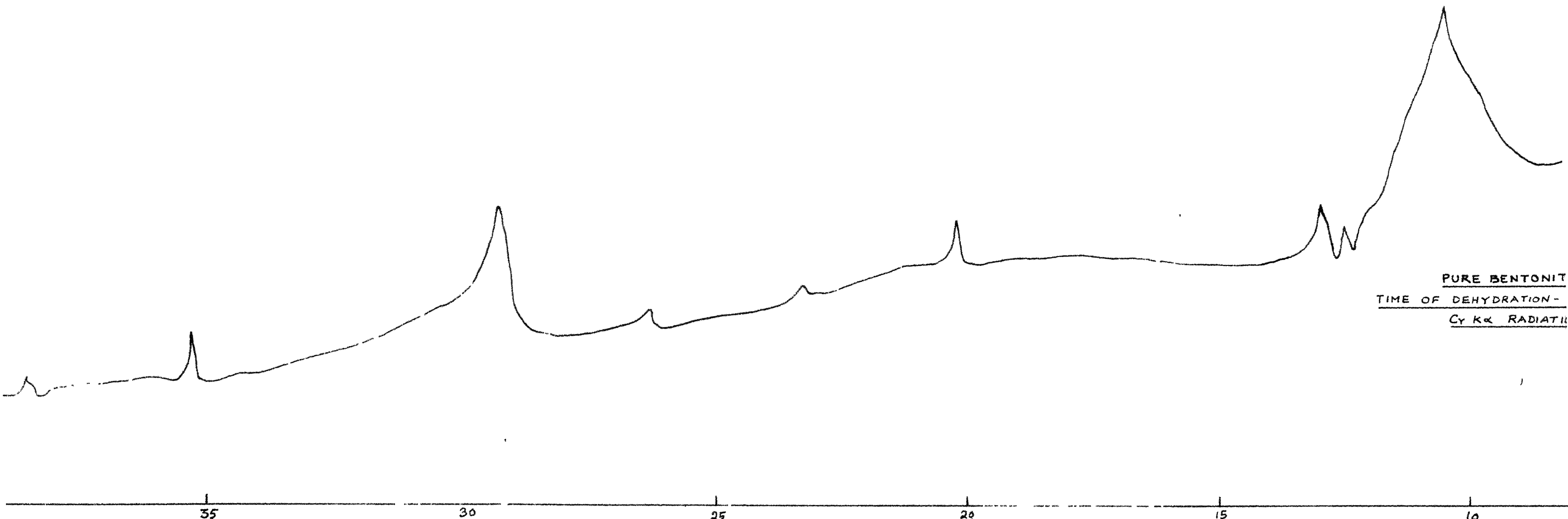
$2\theta^\circ$
FIG 6.4-2



PURE BENTONITE
TIME OF DEHYDRATION - 24 HOURS
Cy K α RADIATION

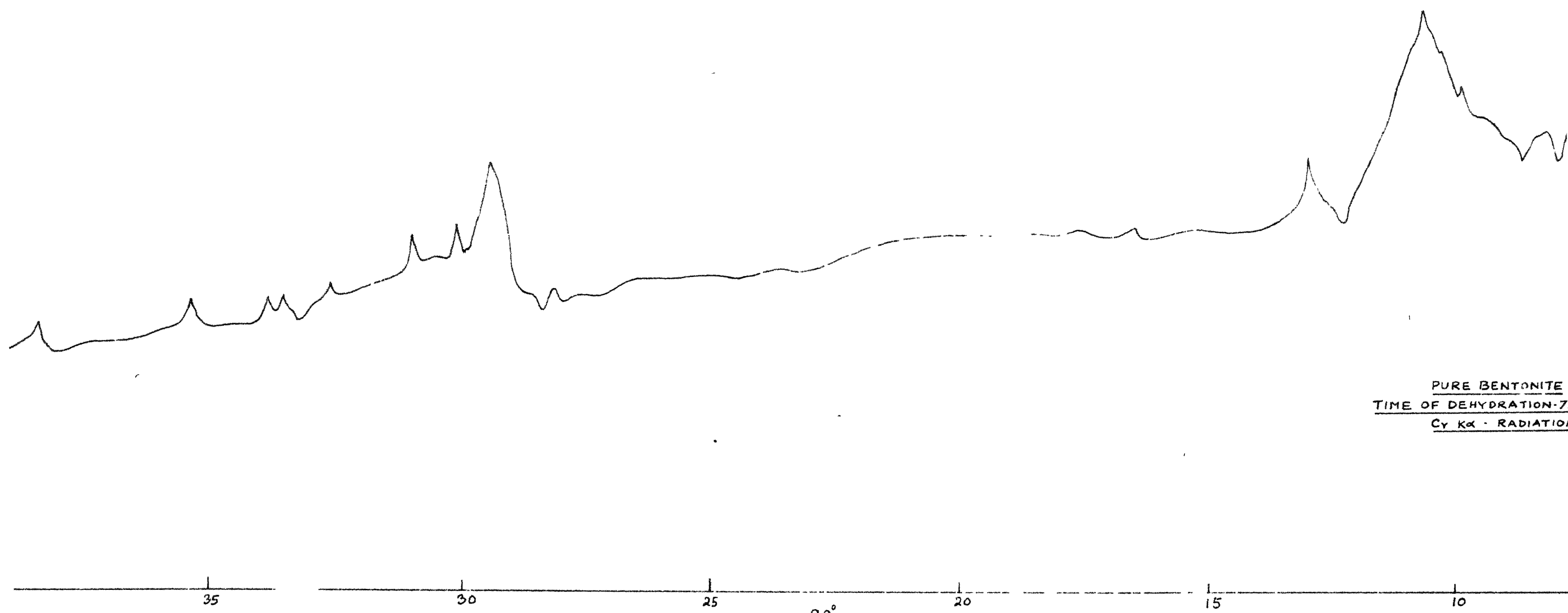
$2\theta^\circ$
FIG 6.4-3





PURE BENTONIT
TIME OF DEHYDRATION -
Cy K α RADIATION

$2\theta^\circ$
Fig G-4-5



PURE BENTONITE
TIME OF DEHYDRATION-72
cy K α - RADIATION

$2\theta^\circ$
Fig 6.4-6

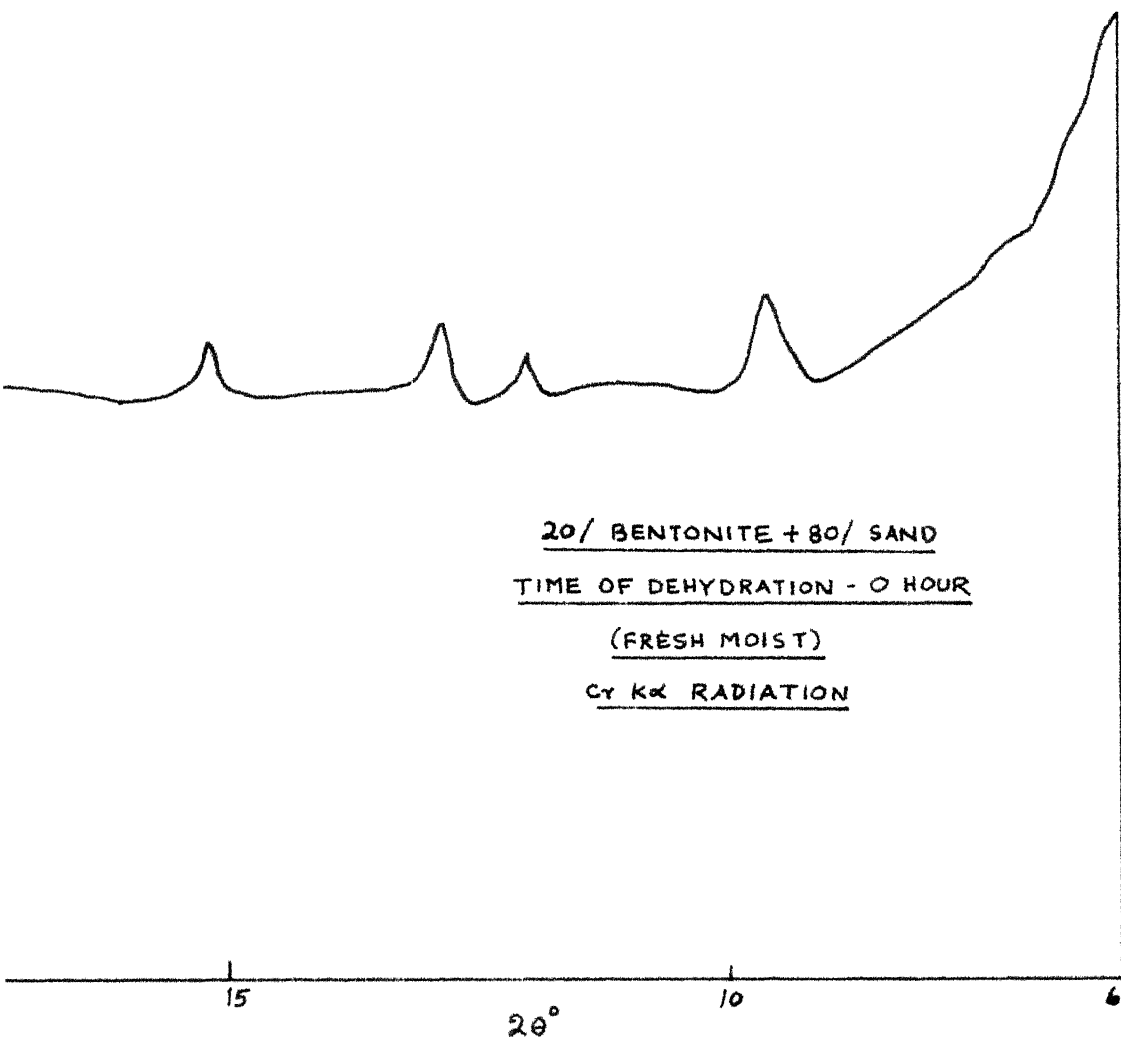


Fig 64-7

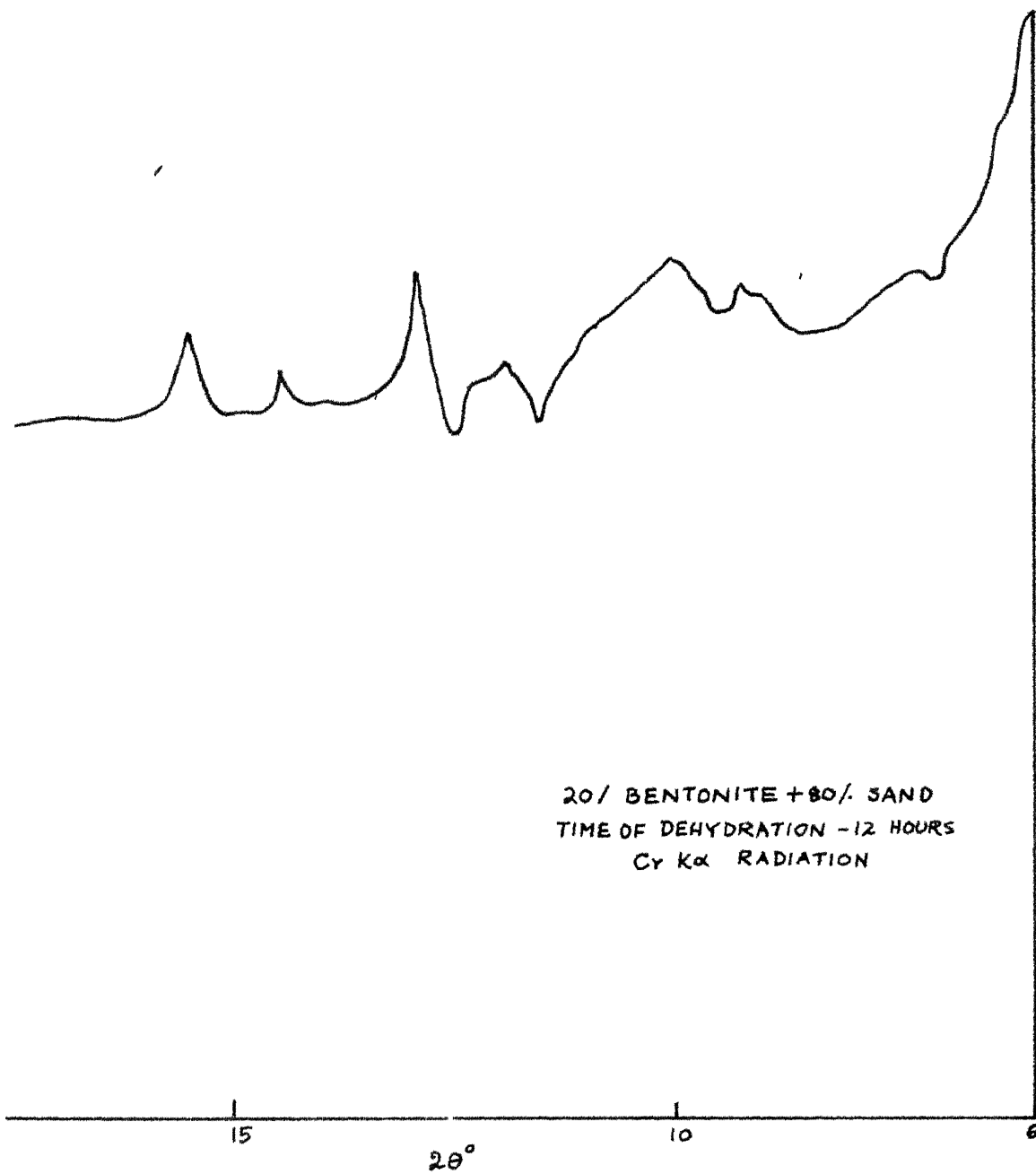


FIG 6 4 - 8

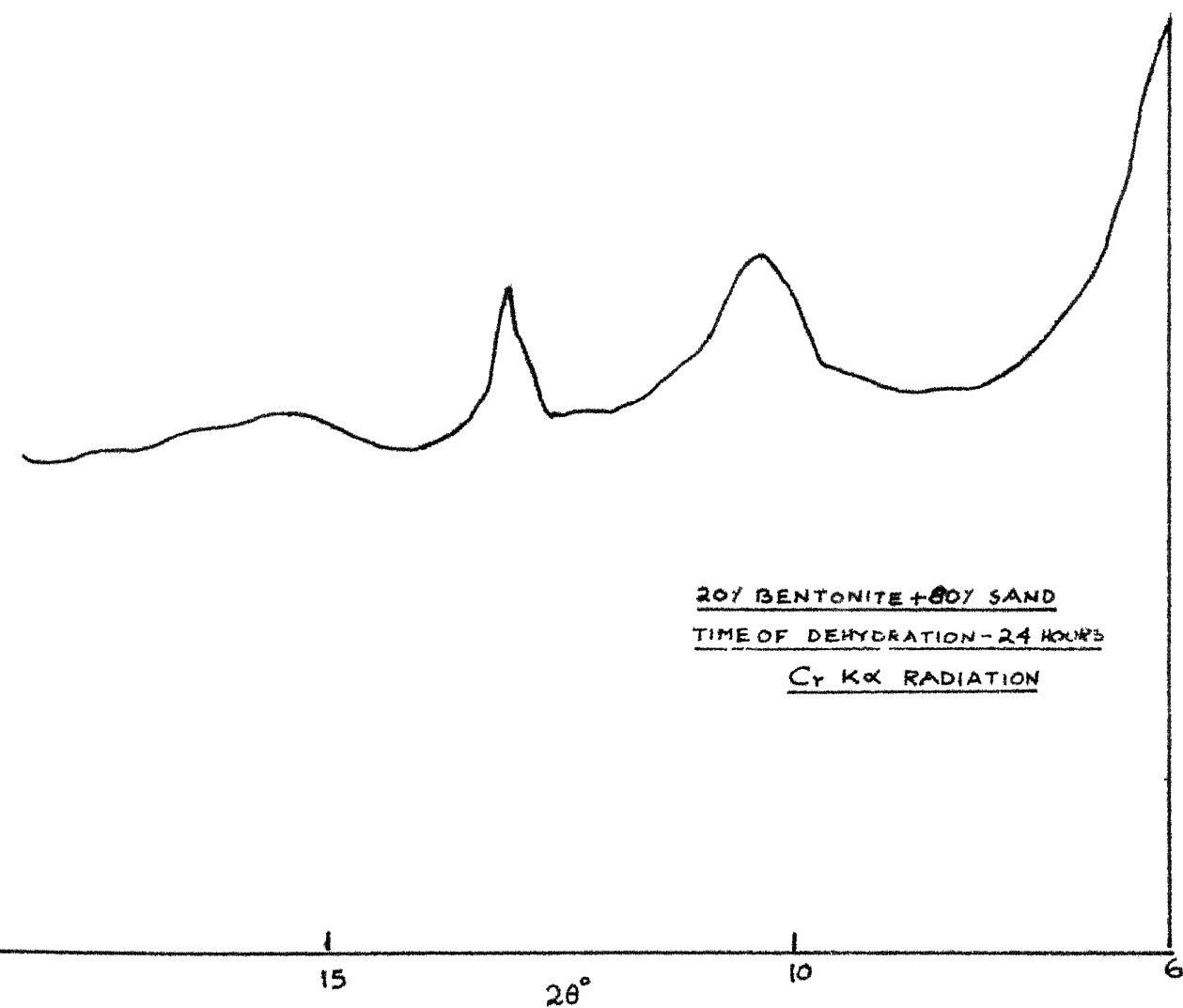
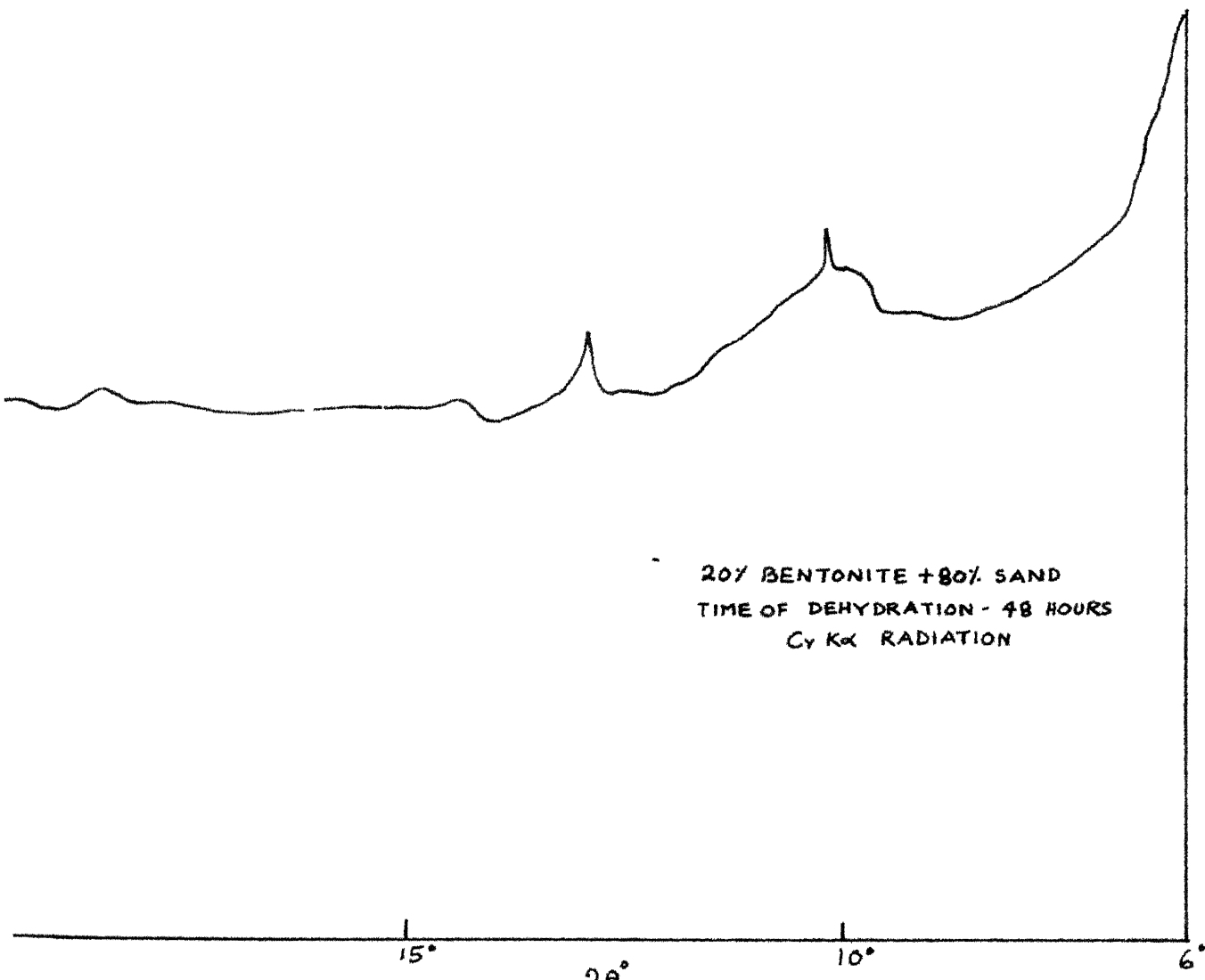


Fig 6.4-9



20% BENTONITE + 80% SAND
TIME OF DEHYDRATION - 48 HOURS
C_K RADIATION

Fig 6-4-11



20/ BENTONITE + 80/ SAND
TIME OF DEHYDRATION - 72 HOURS
Cr K α RADIATION

15 10 6
2 θ°

Fig 6.4-12

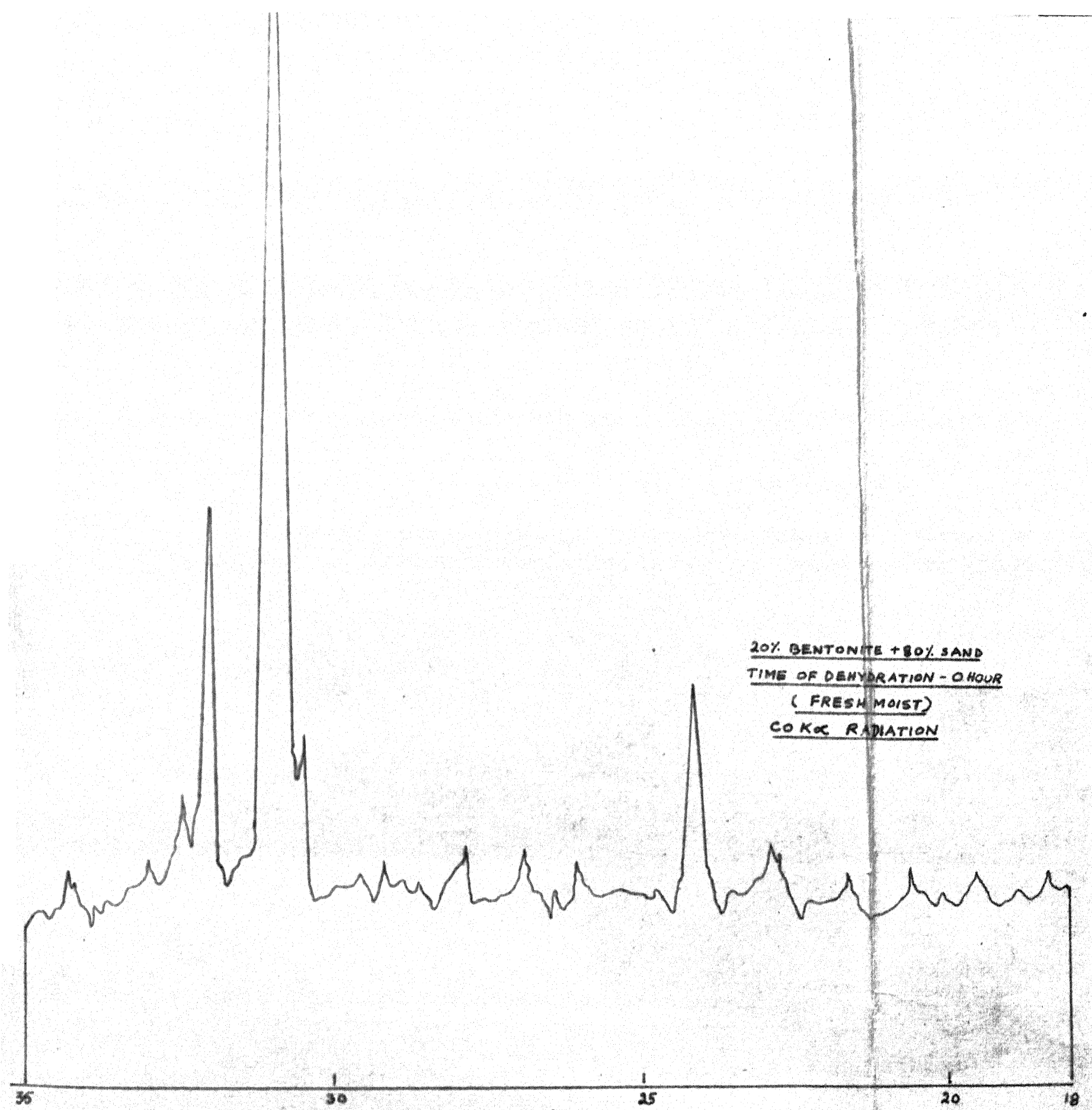
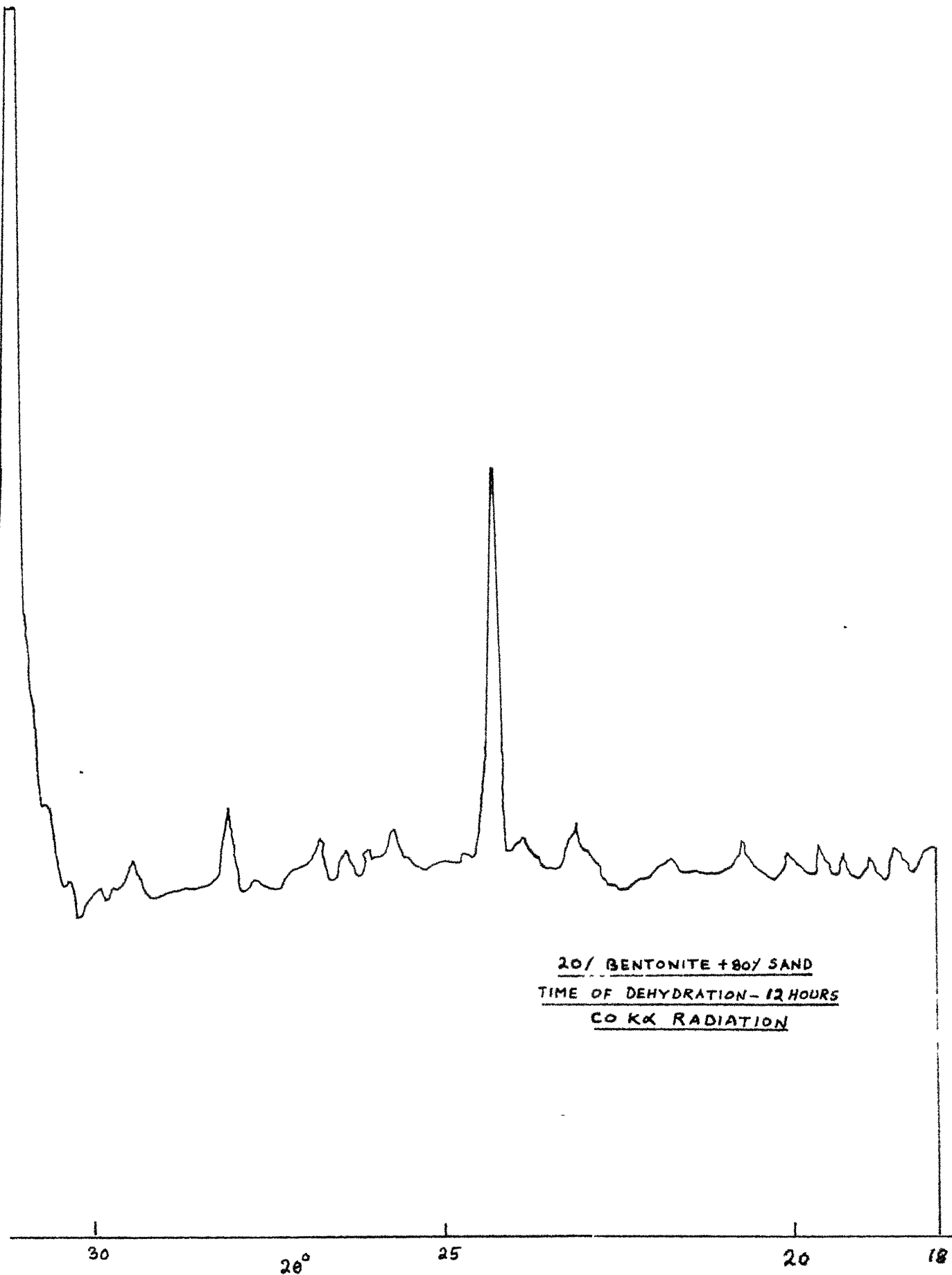


Fig G-4-13



20/ BENTONITE + 80/ SAND
TIME OF DEHYDRATION - 12 HOURS
CO K α RADIATION

Fig 64-14

20% BENTONITE + 80% SAND

TIME OF DEHYDRATION - 36 HOURS

CO K α RADIATION

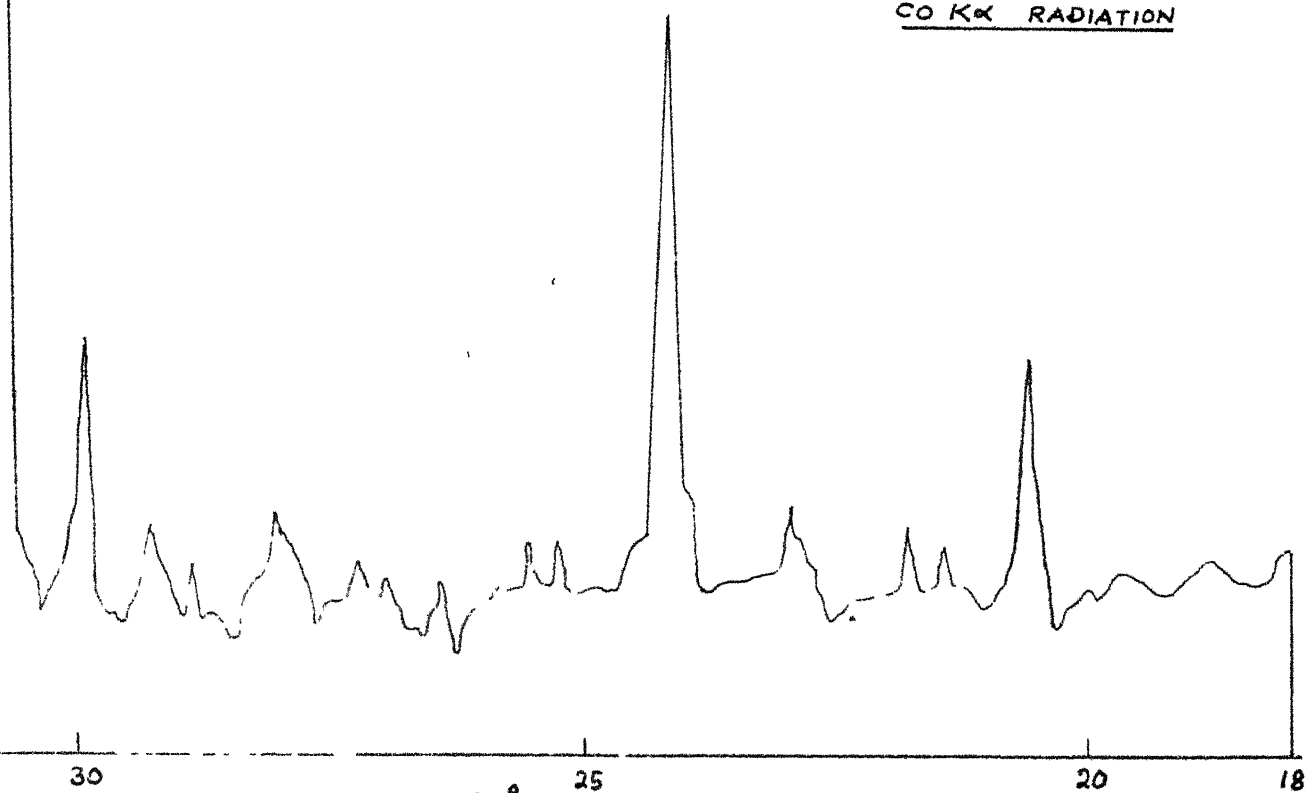
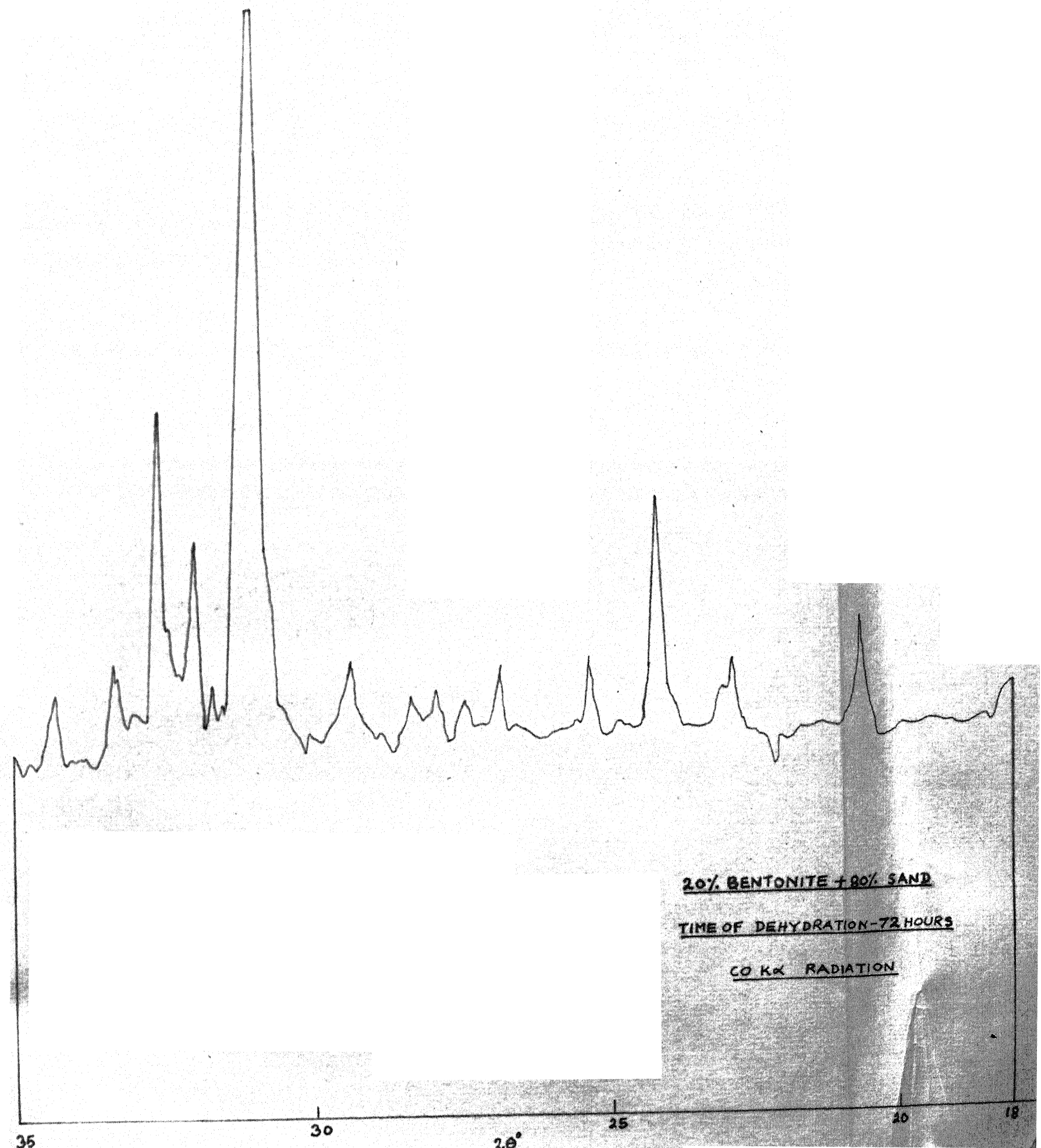


Fig 6 4 - 15



20% BENTONITE + 80% SAND
TIME OF DEHYDRATION - 72 HOURS
CO K α RADIATION

35

30

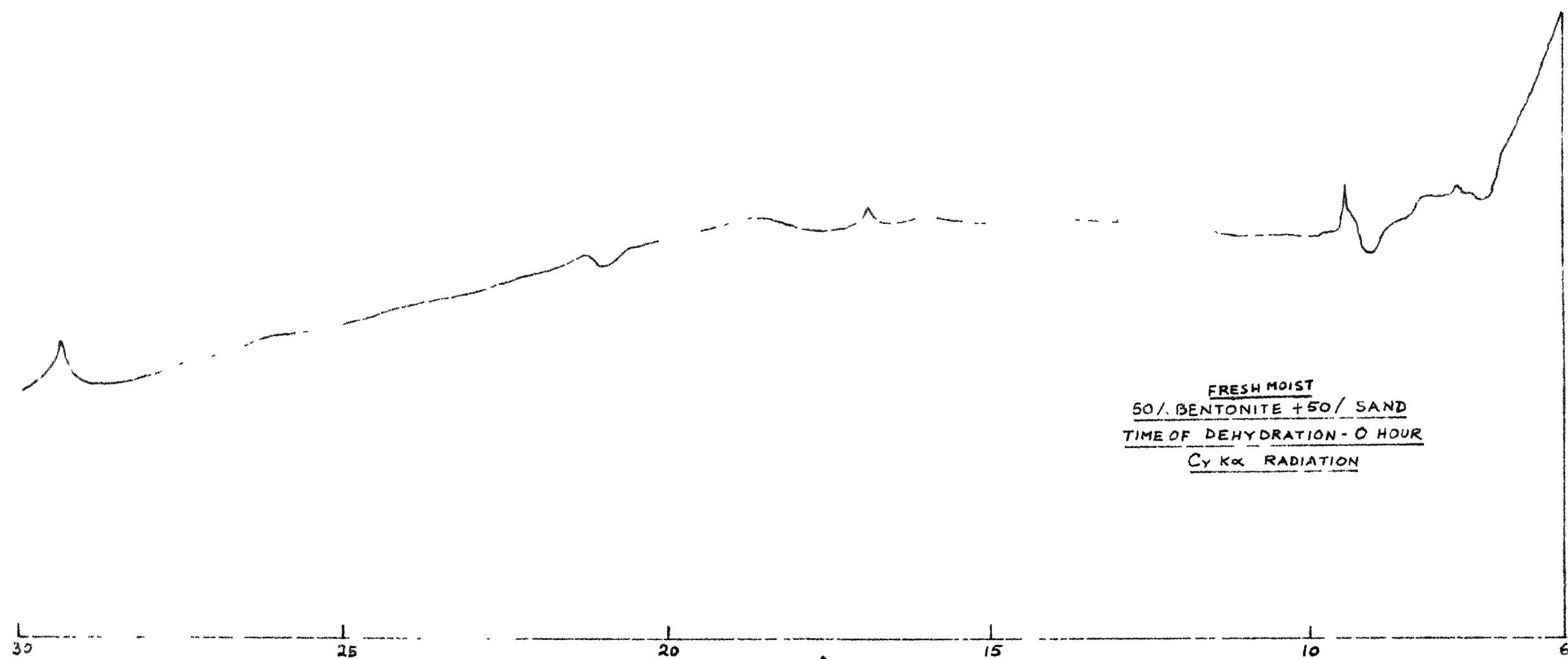
20°

25

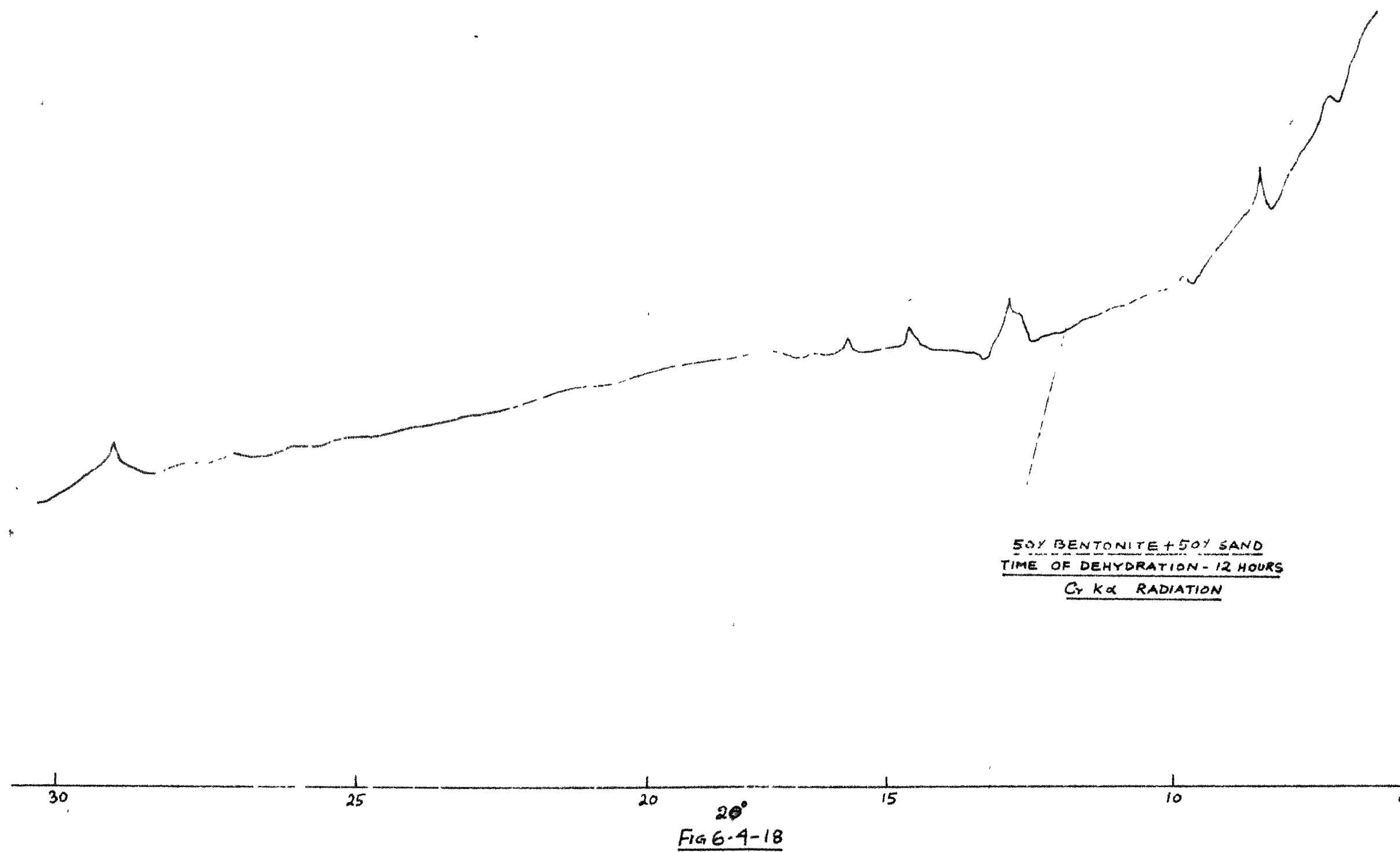
20

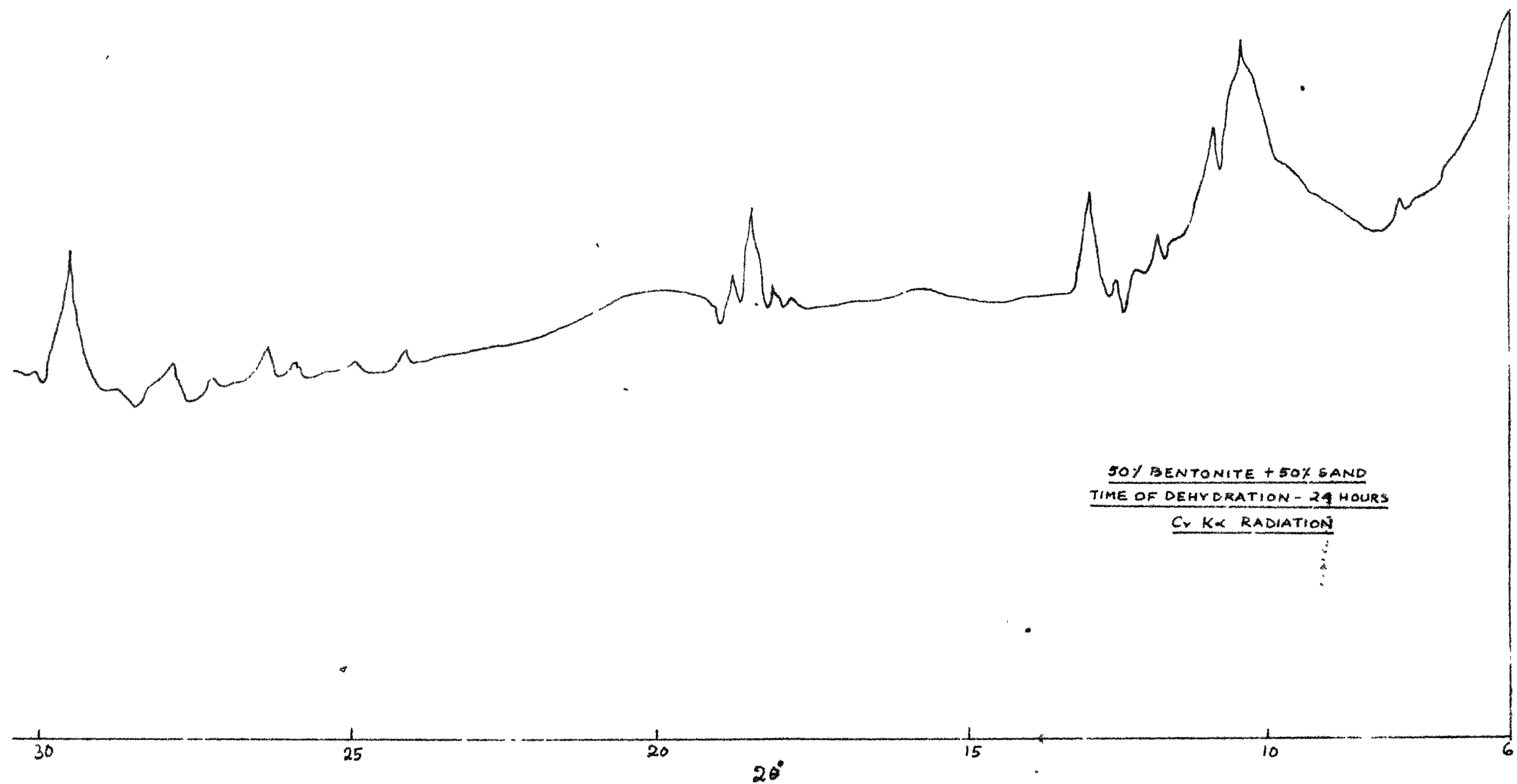
15

Fig 6.4-16

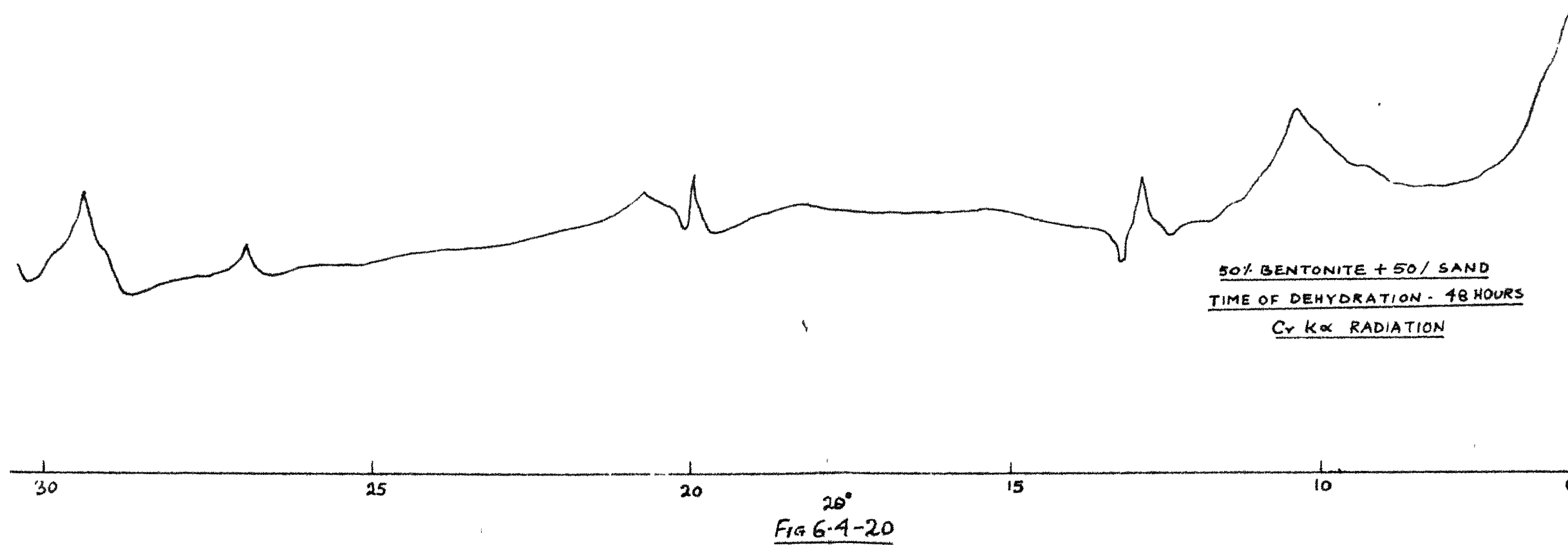


2 θ°
Fig 6 4-17





26
FIG 6-4-19



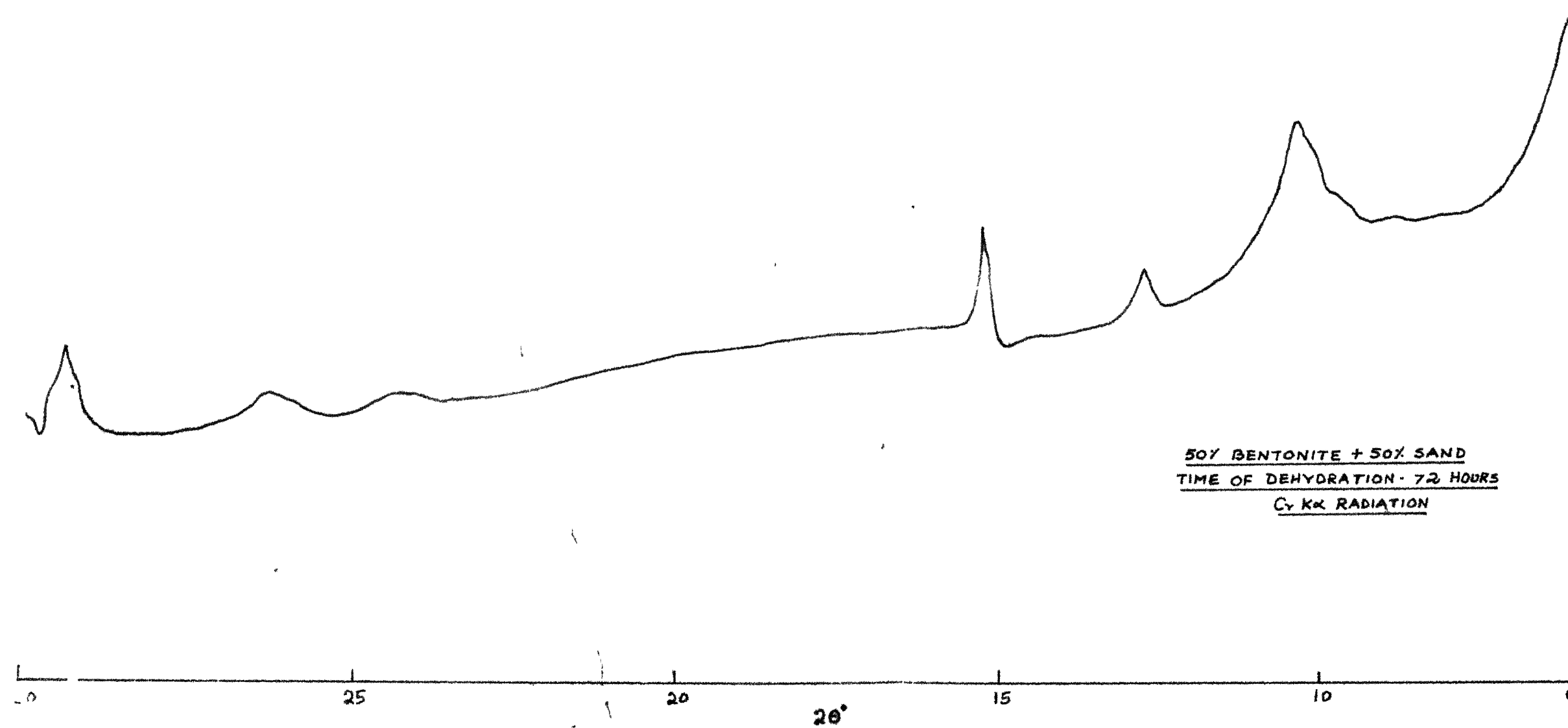


Fig 6.4-21

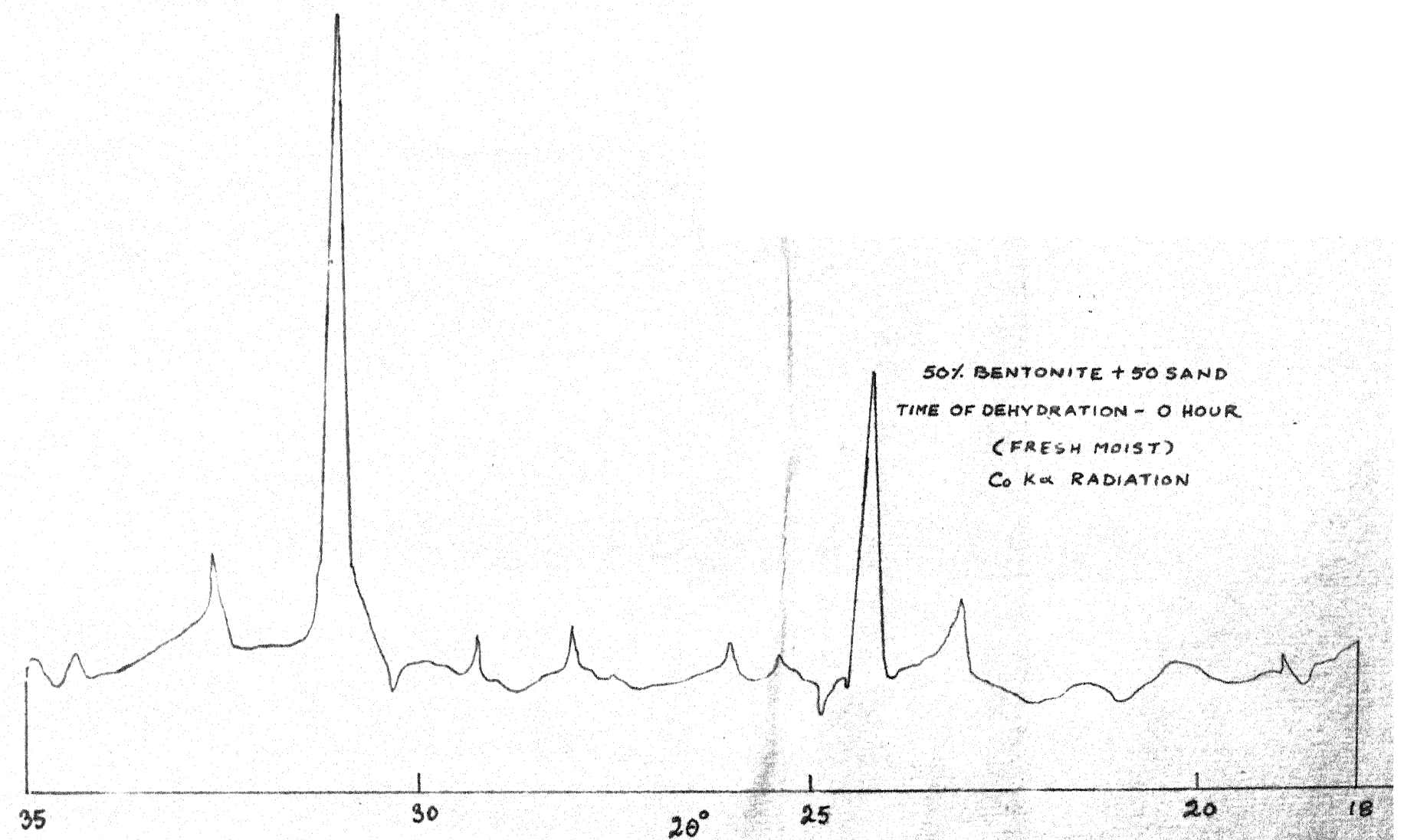
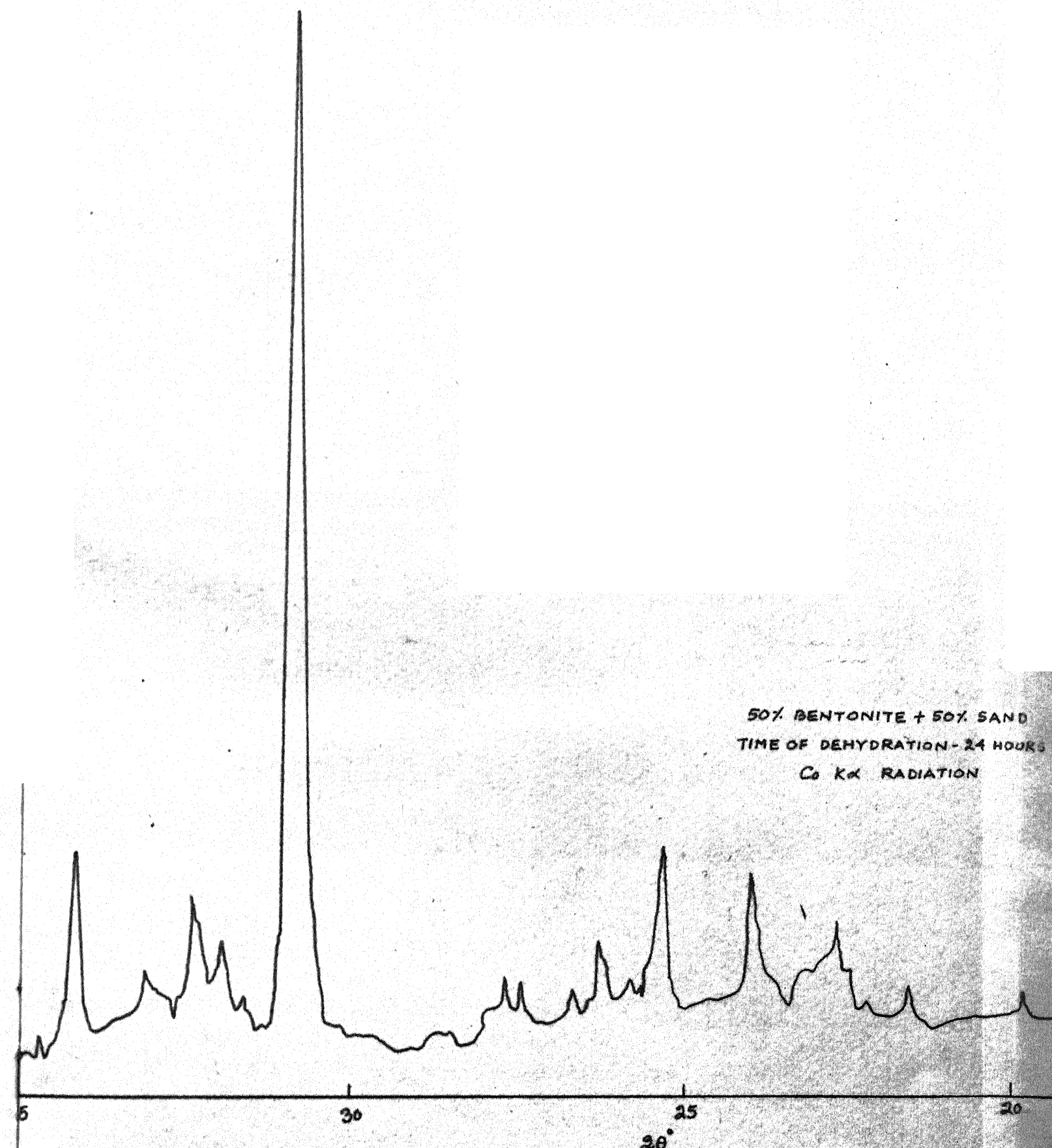
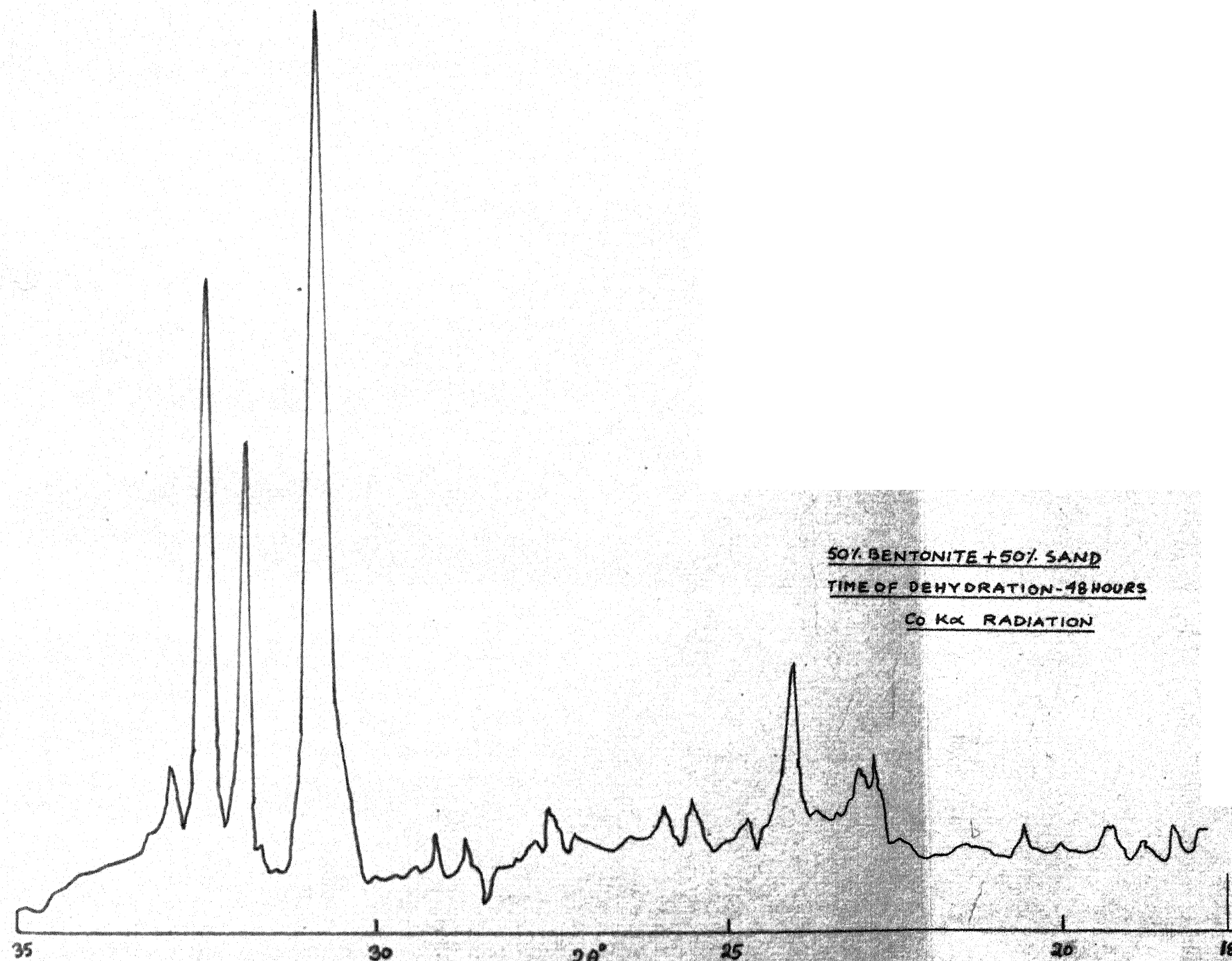


Fig 6-1-22



26°
FIG 6.4-23



50% BENTONITE + 50% SAND
TIME OF DEHYDRATION - 48 HOURS
CO K α RADIATION

Fig 6-4-24

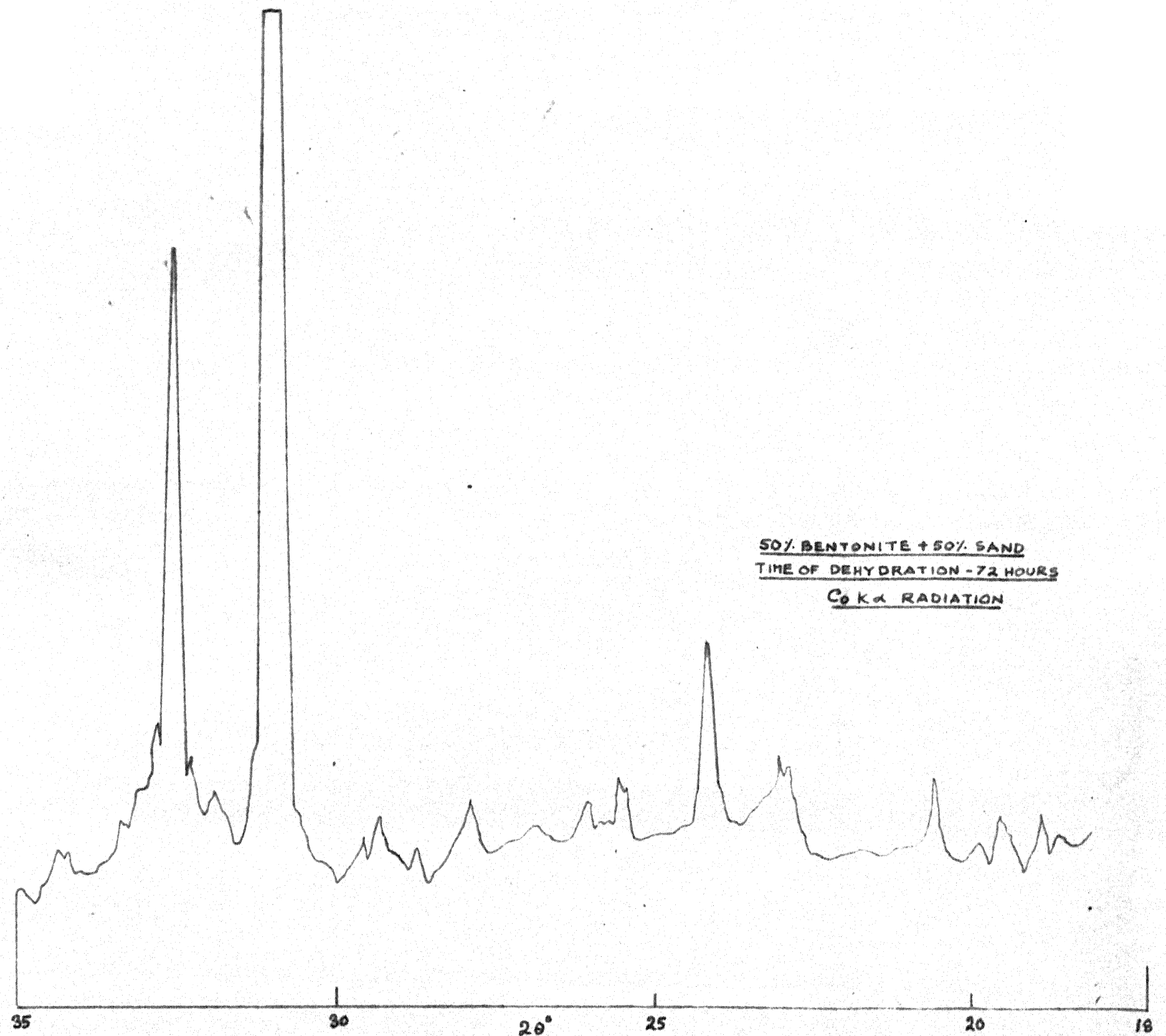


Fig 6.4-25

70% BENTONITE + 30% SAND
TIME OF DEHYDRATION - 0 HOUR
(FRESH MOIST)
Co K α RADIATION

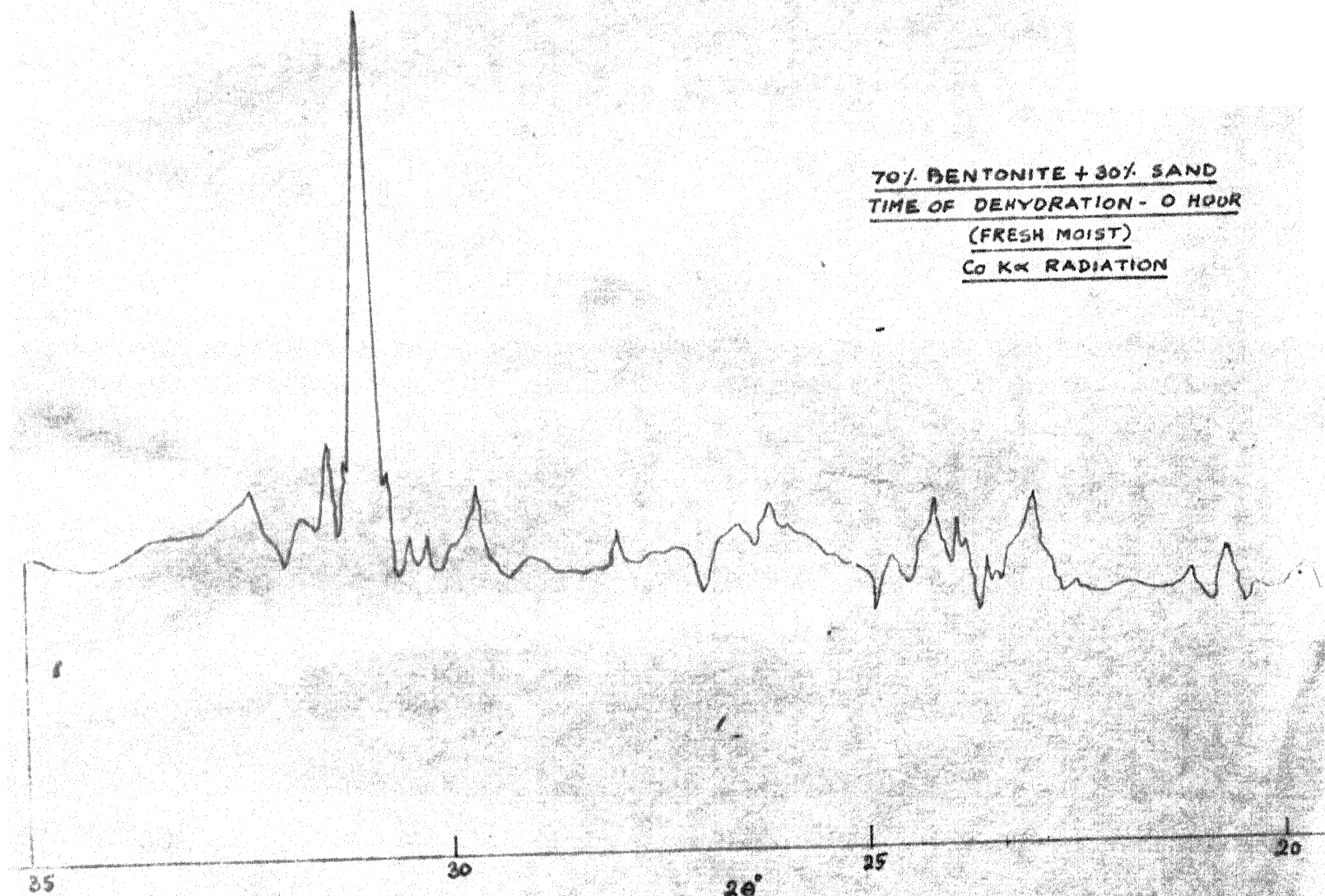
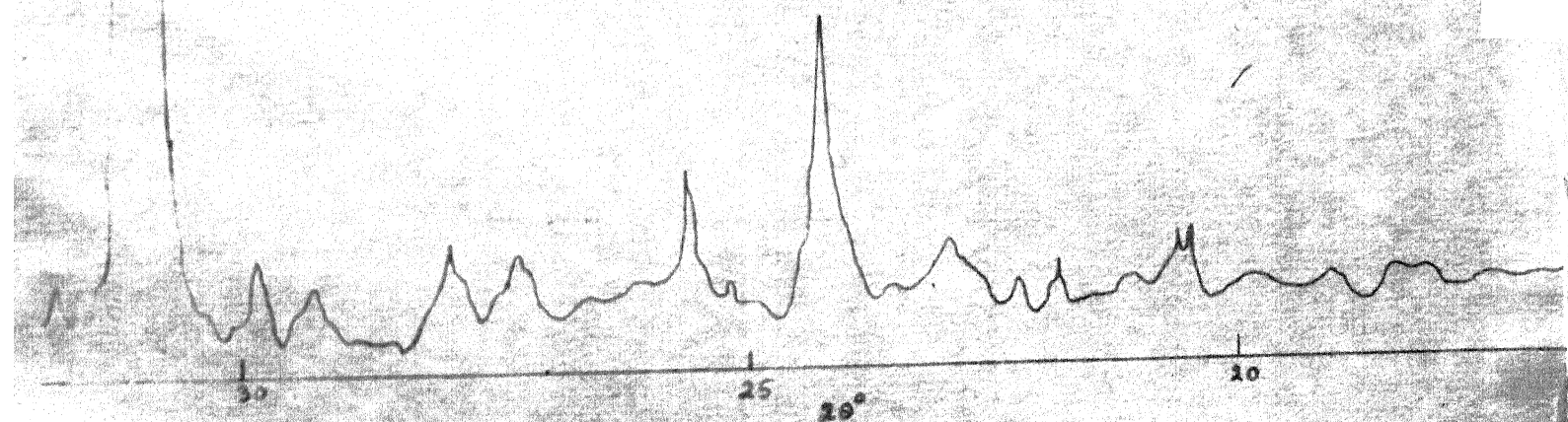


Fig. 6.4-26

70% BENTONITE + 30% SAND
TIME OF DEHYDRATION - 24 HOURS
Co K α RADIATION



6.4.27

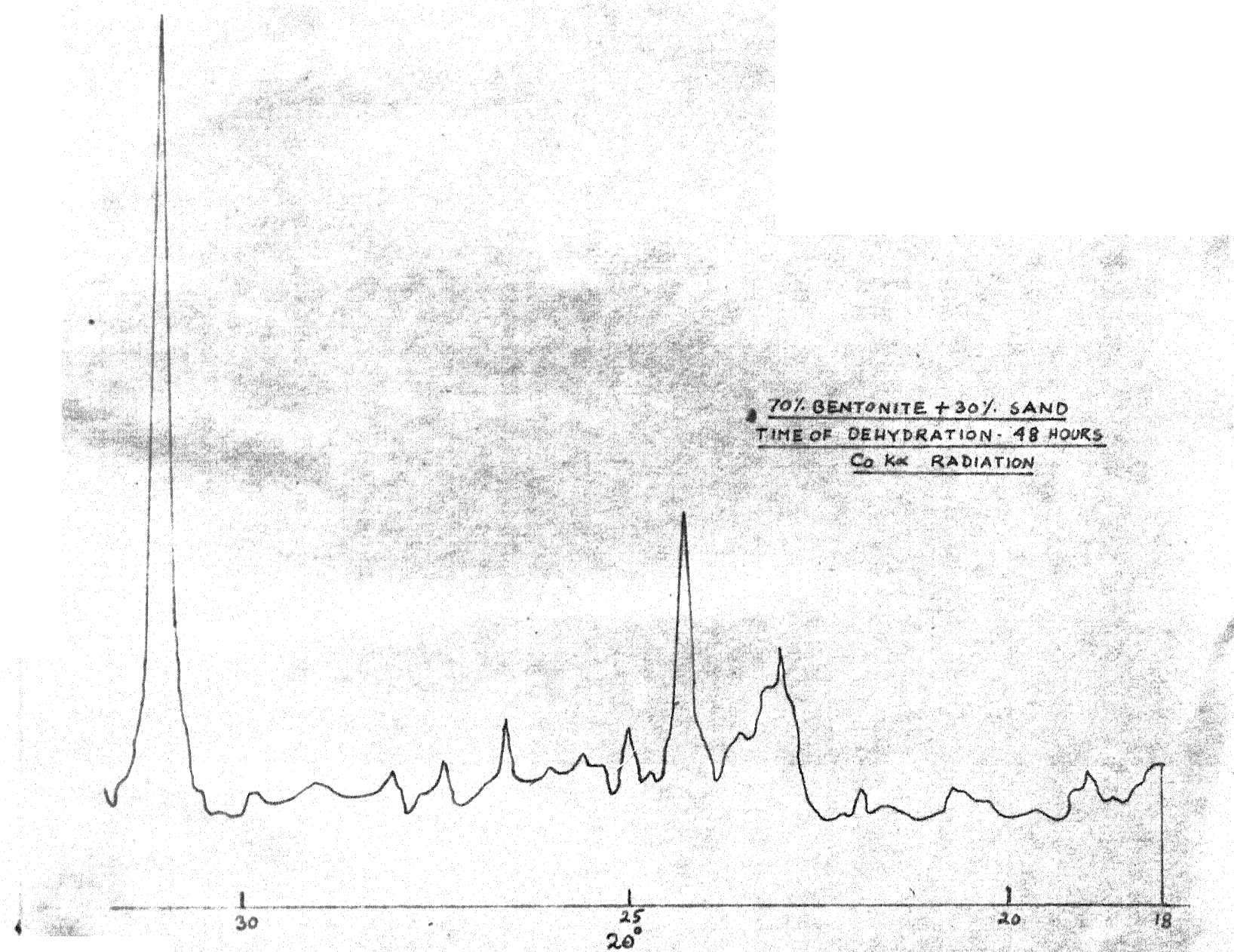
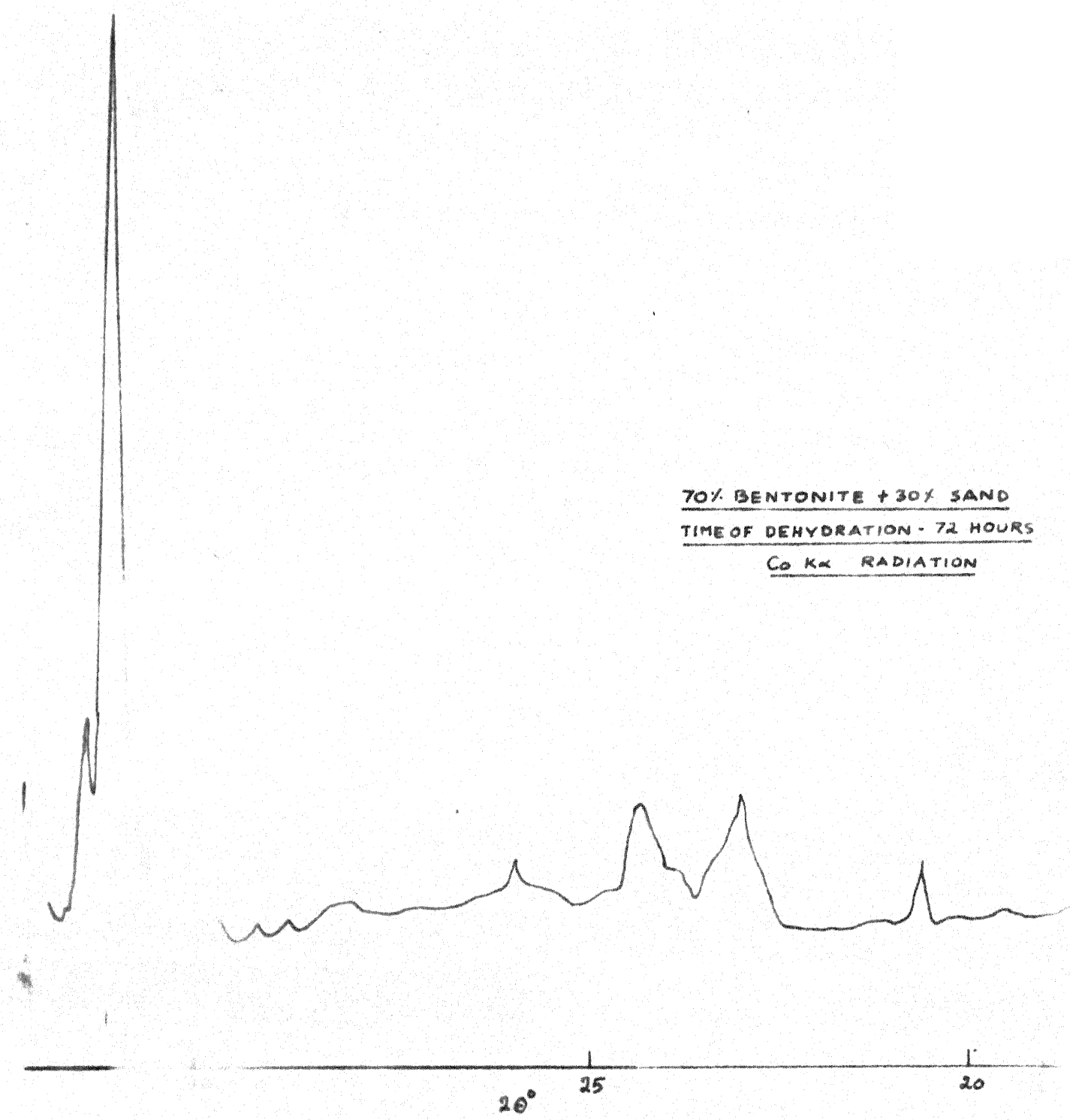


Fig 6-4-28



70% BENTONITE + 30% SAND
TIME OF DEHYDRATION - 72 HOURS
Co K α RADIATION

Fig 6.4 - 29

Thesis

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691.1

326s Das, A K

Studies on certain aspects
of shrinkage and swelling in
clay-sand systems.